

# Industrial Gas Handling

Acid Recovery  
and Recycling

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Flow  
Instrumentation

Facts at Your  
Fingertips:  
Ion-Exchange  
Resins

Focus on  
Milling

Polyester  
Production



June 2020

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# Editor's Page

## Accelerating the WFH trend

The necessity to stay at home during the COVID-19 pandemic has thrust many into working from home (WFH) and setting up workplaces in dining rooms, living rooms, basements or anywhere they could. The new at-home workforce quickly became familiar with video-conferencing and document-sharing software that they may never have used before. Even for those who were already working from home, this time is different. Family members are now at home and everyone is working, studying and socializing in sometimes close living quarters. While the trend before the pandemic was toward an increase in working from home, the current situation has forced an extraordinary change in our work situation, and one that is expected to have lasting effects.

### The challenges

While WFH is often sought-after by employees who want a better work-life balance, it is not preferred by everyone, and it presents challenges. These are exacerbated by the current pandemic, during which even seemingly simple tasks, like getting groceries, can be difficult.

According to Bloomberg<sup>1</sup>, a major VPN (virtual private network) provider reported that during the pandemic, workers in the U.S. used a business VPN an average of three hours more per day than usual — indicating a longer-than-normal workday. Rather than the anticipated gain in free time due to the lack of commuting, workers can actually feel more stress and overworked with the lines between work and personal time blurred. Ironically, while one of the benefits of WFH is seen to be better work-life balance, one of the perhaps most difficult aspects of WFH is the propensity toward a lack of boundaries between work and personal time. And during this pandemic, many feel more pressure at work to do more, as companies face economic stresses. In a recent poll by Eagle Hill Consulting<sup>2</sup>, 45% of U.S. employees said they were experiencing burnout, with one in four attributing it to the COVID-19 pandemic. The top reasons for burnout cited were workload (45%) and juggling professional and personal lives (35%). But still, those working from home feel very fortunate to have jobs as unemployment rates skyrocket.

### Post the COVID-19 crisis

While working from home is far from ideal during this time of crisis, both employees and employers are seeing benefits that will accelerate the trend after the COVID-19 crisis. Last month, Global Workplace Analytics reported findings from a global survey<sup>3</sup> of 2,600 employees taken during the pandemic. Only 6% said they would not want to work from home in the future and 77% said they do want to continue to work from home, at least part-time, after the pandemic.

Companies that were slow to allow employees to WFH, are now witnessing how technology and a willing workforce can make this a viable longterm option. In a survey of 867 global CFOs taken last month, PricewaterhouseCoopers (PwC)<sup>4</sup> found that about half said they are considering making remote working a permanent option where feasible. And they are looking at ways of accelerating automation and other new ways of working.

*Dorothy Lozowski, Editorial Director*

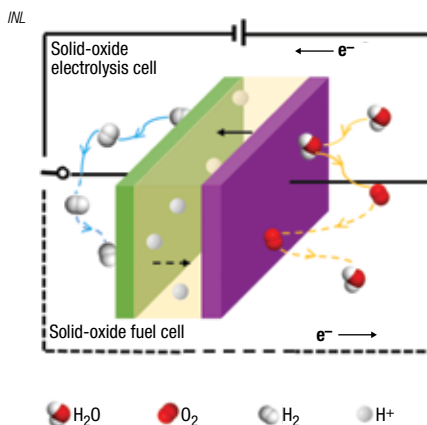


1. Three Hours Longer, the Pandemic Workday Has Obliterated Work-Life Balance, Bloomberg Business, April 23, 2020 www.bloomberg.com
2. www.eaglehillconsulting.com
3. Global Work-From-Home Experience Survey www.globalworkplace-analytics.com
4. www.pwc.com

## This self-sustaining cell makes electrolysis more practical

A team of researchers from Idaho National Laboratory (INL; Idaho Falls; [www.inl.gov](http://www.inl.gov)) has developed a new electrode material that simplifies hydrogen generation and energy storage via protonic, ceramic electrochemical cells (PCECs). Conventional electrolysis technologies use electricity to efficiently split water, but are limited by their extremely high operating temperatures — often as high as 800°C, which make them cost-prohibitive for wide market penetration. Furthermore, at these high temperatures, conductor materials can quickly degrade, explains Dong Ding, INL chemical processing group lead.

The INL team developed a perovskite-based oxygen electrode that not only enables operation at considerably lower temperatures (400–600°C), but also exhibits “triple-conducting” behavior — meaning that it can conduct electrons, oxygen ions and protons within a PCEC. “We doped nickel into conventional praseodymium cobalt oxide, and we observed that this doping strategy reduces proton migration drastically and greatly improves electrokinetics,” adds Ding. The triple-conducting characteristic means that the PCEC can be run reversibly without additional hydrogen fuel. “Starting with steam as the feedstock, this a self-sustaining operation to maintain the switching



between hydrogen production and power generation,” says Ding.

The new material is also considerably simpler to synthesize, since it involves fewer elemental components than typical PCEC electrodes, which may require rare materials or more complex doping processes to manufacture. Ding and his team have already fabricated the new cells in industry-standard sizes for adoption into multicell stacks or modular installations. They are also working to test the material's compatibility with other chemicals to investigate other potential PCEC applications, including the electrochemical activation of natural gas for ethylene and hydrogen co-production.

## Thermal energy-storage system using phase-change materials

Scientists at the U.S. Department of Energy's Argonne National Laboratory (Lemont, Ill.; [www.anl.gov](http://www.anl.gov)) have developed a thermal energy storage system for industrial processes that can capture and store typically wasted heat for later use. Originally conceived as a way to store surplus heat from concentrated solar power installations, the system is being refined for other applications, including storing heat from industrial combustion processes, solar-powered desalination plants, combined heat and power (CHP) facilities and heavy-duty trucks.

The device stores and releases latent heat using specific types of molten salts as phase-change materials that melt when heat is captured from a process, then solidify when the heat is released for later use. In the development of the storage system, the Argonne team, led by senior materials scientist Dileep Singh, overcame a key limitation of molten salts. While molten salts can be effective as phase-change materials for re-

taining heat, they are typically poor thermal conductors, “so it takes too long for them to absorb and release energy,” the team says.

To overcome this, the researchers devised a proprietary method to integrate the phase-change materials with another high-thermal-conductivity material. The composite material system is sealed into a cylindrical module and bathed in an inert gas. Heat stored in the modules can be used to heat water to create steam, for example.

The thermal storage devices can be made in manageable sizes, such as that of a 55-gal drum, and they are modular in nature, Singh says, so they can be scaled easily by adding more of the modules, depending on the application.

The researchers have demonstrated that the system to work at temperatures over 700°C (1,292°F), and are working to integrate it into commercial CHP units. They are also working with various industry partners to adapt the device for different applications.

Edited by:  
**Gerald Ondrey**

### BIOFERTILIZER + BIOGAS

Three new biofertilizer-biogas facilities will be built in the Zachodniopomorskie region of Poland. The plants are being built by Ductor Oy (Helsinki, Finland; [www.ductor.com](http://www.ductor.com)), a Finnish-Swiss biotechnology company funded by Esperotia Investments Ltd.

The plants will use 100% poultry waste to create two separate products, renewable electricity and organic nitrogen fertilizer. This will significantly reduce greenhouse gas emissions from both the energy and agriculture sectors. Two of the new plants will have installed capacity of 0.5 MW and the third will be 1 MW. All three will be operational in 2021.

The three facilities will use 50,000 tons, or about 1% of the total poultry manure produced in Poland each year. Ductor currently has similar projects underway, including five to ten new facilities in Poland and a solid portfolio of projects under development in Europe and the Americas. The system was patented in 2015, and the first operational biofertilizer-biogas facilities opened at the end of last year in Mexico.

### ‘GREEN METHANOL’

As part of the “Power-to-Methanol — Green Methanol” project, the Fraunhofer Institute for Solar Energy Systems (Freiburg, Germany; [www.ise.fraunhofer.de](http://www.ise.fraunhofer.de)) recently commissioned a miniplant for synthesizing methanol from H<sub>2</sub> and CO<sub>2</sub>. Featuring a measurement technique with high temporal and spatial resolution, the setup enables research on methanol synthesis, among other things, within the framework of power-to-liquid (PtL) processes on an industrial scale. Here, the main focus of the research is on dynamic reactor operation and unconventional gas compositions using H<sub>2</sub> produced by

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electrolysis and gas streams containing CO<sub>2</sub>.

Conventional production processes based on fossil feedstock have reached technical maturity over the past decades, but have large CO<sub>2</sub> emissions. "In contrast, methanol synthesis in PtL processes offers the potential to bind CO<sub>2</sub> from biomass, for example, and to reuse it," explains Achim Schaadt, head of the Thermochemical Processes department at Fraunhofer ISE.

A number of questions still need to be answered before this process can be implemented on an industrial scale. For example, a high CO<sub>2</sub> content in the synthesis gas lead to accelerated aging of the catalyst and to lower chemical yields. Furthermore, fluctuations both in the amount of H<sub>2</sub> produced by renewables and in the process for the provision of CO<sub>2</sub> may require dynamic synthesis operation.

The project is funded by the German Federal Ministry for Economic Affairs and Energy and led by Dechema e. V., with industrial partners CropEnergies AG, Clariant AG and thyssenkrupp Industrial Solutions AG.

## METAL AEROGELS

A new class of porous materials, noble metal aerogels (NMAs) have drawn interest because of their useful properties, including self-supported architectures, high surface areas and numerous optical and catalytic active sites. However, current production methods suffer from long fabrication times, unavoidable impurities and uncontrolled multiscale structures.

Now, chemists from the Dresden University of Technology (Germany; [www.tu-dresden.de](http://www.tu-dresden.de)) have developed a freeze-thaw method capable of synthesizing various NMAs that have clean surfaces and multiscale structure. Because of their hierarchical structures and unique optical properties, the NMAs exhibit "outstanding" performance for the electro-

## Filtering out and detoxifying Cr(VI) from water

Chemists from the Swiss Federal Institute of Technology Lausanne (EPFL; Switzerland; [www.epfl.ch](http://www.epfl.ch)) have developed "sponges" designed to capture various target substances, such as gold, mercury and lead, dissolved in solution. Now, they have developed one for capturing toxic hexavalent chromium from water. The material — a composite bead of a metal organic framework (MOF) on a polymer — not only has a high adsorption capacity for Cr(VI), but it also acts as a photocatalyst, whereby the Cr(VI) is converted to the less toxic Cr(III).

As described in an article published last month in the *Journal of Materials Chemistry A*, the adsorbent sponge is made by first functionalizing a known Zr-MOF, UiO-66, with double amino groups. This modification permits the new material, Zr-BDC-(NH<sub>2</sub>)<sub>2</sub>, to serve a dual-purpose as both adsorbent and photocatalyst, according to the chemists. Next, Zr-BDC-(NH<sub>2</sub>)<sub>2</sub> was incorporated into MOF@



polymer beads using polyethersulfone (PES) that was chemically modified with carboxylic acid groups to improve hydrophilicity.

The researchers have demonstrated that the MOFs can extract approximately 208 mg of Cr(VI) per gram of MOF [photo before (left) and after (right)]. Also, shining light on the loaded MOF then transforms the highly toxic Cr(VI) into a relatively nontoxic Cr(III). Further developments are required in order to implement the technology for decontaminating water outside of the laboratory.

Hexavalent chromium continues to contaminate water sources around the world, with one U.S. company fined just this past February for having put employees at risk. Cr(VI) is considered to be extremely toxic, especially when inhaled or ingested, and its use is regulated in Europe and in many countries around the world. It is thought to be genotoxic, leading to DNA damage and the formation of cancerous tumors.

## New process for safer and continuous magnesium production to be piloted

Because of its abundance, light weight and easy alloying, magnesium metal has great potential for components in the automotive, aerospace and military industries. However, the two existing production processes for Mg — the Pidgeon process from ore and electrolytic process from MgCl<sub>2</sub> — both have major drawbacks with safety and environmental impact.

Now, a pilot plant currently in the design phase will demonstrate a new Mg production process that is continuous, rather than batch, and that dramatically reduces the safety and environmental problems compared to existing processes. The process developer, Western Magnesium Corp. (Vancouver, B.C.; [www.westmagcorp.com](http://www.westmagcorp.com)), says the safety, environmental and economic profile of the new process could make Mg production viable in North America. Currently, 85% of Mg comes from China.

The Western Magnesium process begins with mined dolomite (calcium-magnesium carbonate), which is crushed and calcined (thermal decomposition) to make MgO. The MgO is combined with ferrosilicon, a reductant that is also used in the Pidgeon process, and a catalyst, and formed into briquettes. The briquettes are fed into the top of a newly

designed continuous reactor, where they are heated, allowing the silicon to react with oxygen and reduce the magnesium to metal. The vaporized Mg is collected and removed from the reactor as molten Mg using a proprietary Mg-recovery system that was designed specifically for this process.

"In the Pidgeon process, which occurs batch-wise, repeated heating and cooling cycles are needed to remove the Mg and drive the reaction, so the process is highly energy-intensive — 40 MWh/ton of Mg," explains Paul Sauvé, vice president of operations at Western Magnesium. "The continuous nature of our process eliminates the temperature cycles, so much less energy is required." To further raise efficiency and reduce environmental footprint, the company's process uses an electric-powered calciner, heat-recovery systems and a method for recovering food-grade CO<sub>2</sub> from the calcination step. Finally, in addition to Mg, the process yields dicalcium silicate, which can be sold as an aggregate material.

Western Magnesium plans for the pilot plant operation to be complete in Q3 2021. The pilot will inform the design of the full-scale plant. After some refinement, the plant will be capable of producing 99.8% pure Mg in ingots, granules or as molten material, Sauvé says.

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## A new trace-moisture analyzer harnesses the power of MOFs

The presence of moisture — even in very small amounts — can be extremely detrimental to the manufacture of many products, including semiconductors. Removal of moisture from a contaminated process is a labor-intensive process that requires large volumes of high-purity gases. Harnessing the optical characteristics of a metal-organic framework (MOF) material, Taiyo Nippon Sanso Corp. (TNSC; Tokyo; [www.tn-sanso.co.jp](http://www.tn-sanso.co.jp)) has developed a new trace moisture analyzer for nitrogen gas that is said to be more compact, sensitive, reliable and faster than current technologies for industrial-gas moisture analysis.

Through joint research with the Faculty of Advanced Science and Technology at Kumamoto University (FAST; [www.fast.kumamoto-u.ac.jp](http://www.fast.kumamoto-u.ac.jp)), TNSC investigated the porous MOF material Cu-BTC, which con-

sists of a divalent copper ion ( $\text{Cu}^{2+}$ ) and benzene-1,3,5-tricarboxylate (BTC). The Cu-BTC MOF's wavelength absorbance changes in conjunction with moisture absorption and desorption, and the analyzer detects this absorbance change and converts it into a moisture-concentration reading. According to TNSC, the new analyzer boasts a response speed of less than five minutes and a lower detection limit of 10 parts per billion by volume (ppbv). The strong correlation demonstrated between changes in wavelength absorption and moisture mean that the MOF-based measurements are highly reliable, with much faster response times than conventional analyzers. TNSC is currently testing the technology at its plants and plans to commercialize the analyzers during the next fiscal year. They are also evaluating analysis of additional gases beyond nitrogen.

oxidation of ethanol. The research, described in a recent issue of *Angewandte Chemie International Edition*, provides new ideas for designing various gel or foam materials for high-performance electrocatalysis and photoelectrocatalysis in fuel cells.

In previous studies, the research group had found unusual self-healing properties of noble metal gels. Inspired by this phenomenon, a freeze-thaw method was developed as an additive-free approach to directly destabilize various dilute metal-nanoparticle solutions. Upon freezing, large aggregates are generated due to the intensified salting-out effects incurred by the dramatically raised local solute concentration. Meanwhile, the aggregates are shaped at micrometer scale by in-situ-formed ice crystals. After thawing, aggregates assemble into monolithic hydrogels as a result of their self-healing properties. Purified and dried, clean hydrogels and the corresponding NMAs are obtained.

The resulting gold-palladium (Au-Pd) aerogels were found to display “impressive” light-driven photoelectrocatalytic performance, delivering a current density of up to 6.5 times higher than that of commercial palladium-on-carbon (Pd/C) for the ethanol oxidation reaction.

### CHITIN FROM SHELLS

Chitin is used in the food industry as thickeners and stabilizers, as well as in anti-microbial food packaging. Conventional routes chemically extract the chitin from

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marine waste, which is costly, consumes large amounts of energy and generates by-products that could be discharged into industrial wastewater.

Now, scientists at Nanyang Technological University, Singapore (NTU Singapore; [www.ntu.edu.sg](http://www.ntu.edu.sg)) have developed a “greener” way to make chitin, by using two forms of food waste — prawn shells and discarded fruit — and fermenting them.

The NTU team tested ten sources of common fruit waste, such as white and red grape pomace, mango and apple peels and others, in fermentation experiments. They found that fruit waste contained enough sugar content to power the fermentation process that breaks prawn shells down into chitin.

The fermentation process using the sugar content from the fruit waste produced higher quality chitin than the commercial one. Using X-ray diffraction, the extracted crude chitin from the fermentation process had a crystallinity index of 98.16%, which indicates a higher quality than the 87.56% index observed from commercial chitin samples.

## NOX ADSORPTION

A new filtration system that adsorbs harmful oxides of nitrogen (NOx) from ambient air has been successfully tested and is now ready for scaleup. Produced by Krajete GmbH (Pasching, Austria; [www.krajete.com](http://www.krajete.com)), the system was installed in Heilbronn, Germany, where it operated for more than a year without the need for maintenance, filtering 1,500 m<sup>3</sup>/h of air. The next size will have a capacity that is more than 15 times higher.

The technology uses aluminum silicates that bind NOx under certain conditions. “What sets our technology apart is that the filter material adsorbs NOx at both very high and very low NOx concentrations,” says company founder and CEO Alexander Krajete. This is a fundamental requirement that needs to be met if the technology is to be used to clean ambient air (with relatively low NOx concentration) and to purify vehicle exhaust fumes (with high NOx concentrations), he says.

In response to the positive re-

## Progress on a longer-lasting catalyst for coke-free CO<sub>2</sub> reduction

One of the main problems in the conversion of greenhouse gases, such as carbon dioxide and methane, into liquid fuel or hydrogen is the development of coke- and sintering-resistant catalysts. Nickel on magnesium oxide (Ni/MgO) has long been identified as a suitable catalyst, but rapid coke formation and sintering have prevented its use at an industrial scale.

Now a group of researchers claims to have overcome those problems. The group, led by professor Cafer T. Yavuz of the Korea Advanced Institute of Science and Technology (Daejeon, South Korea; [www.kaist.ac.kr](http://www.kaist.ac.kr)), includes people from Gebze Technical University (Gebze, Turkey), Saudi Aramco (Dhahran, Saudi Arabia), and Pohang Accelerator Laboratory (Pohang, South Korea).

The researchers reported a molybdenum-

doped nickel nanocatalyst that is stabilized at the edges of a single-crystalline MgO support. As the ingredients were heated under reactive gas, the nanoparticles moved on the crystal surface seeking anchoring points. The resulting activated catalyst sealed its own high-energy sites and permanently fixed the location of the nanoparticles. Thus, the nickel-based catalyst would not have carbon buildup, nor would the surface particles bind to one another.

According to the group, the catalyst runs more than 850 h of continuous operation under 60 liters per unit mass of catalyst per hour reactive gas flow with no detectable coking. Synchrotron studies also showed no sintering and revealed that during activation, 2.9-nm synthesized crystallites move to combine into stable 17-nm grains at the edges of MgO crystals.

## Anti-fouling pre-treatment for membranes

Researchers at the Korea Institute of Science and Technology (KIST; Seoul, South Korea; [www.kist.re.kr](http://www.kist.re.kr)) led by Seongpil Jeong have developed a pre-treatment method for membrane distillation desalination processes that involves adding magnesium to seawater to prevent scale formation on the membrane. Adding magnesium chloride to the seawater before contact with the membrane allows Mg ions to combine with sulfate and carbonate ions present in the seawater, and thus prevent formation of calcium carbonate and calcium sulfate, which

were found to be the major components of scale, the researchers say.

The team also found that the added MgCl<sub>2</sub> increased the stability of the hydrophobic membrane, thereby increasing wetting resistance. Membrane distillation desalination technology involves heating seawater to generate a vapor, which is passed through a hydrophobic membrane before condensing as fresh water. Fouling and wetting can lengthen production times for freshwater and shorten the lifespan of the membrane in the distillation process, thereby increasing the costs of freshwater production.

## An alternative crop for bioethanol

The *Agave tequilana* plant, which is native to Mexico and used to make the popular drink tequila, promises significant advantages over sugarcane and corn as a source of bioethanol. The *Agave* plant is now being grown as a biofuel source on the Atherton Tablelands in North Queensland by MSF Sugar (Gordonvale, Australia; [www.msfsugar.com.au](http://www.msfsugar.com.au)).

Studies of the plant's lifecycle and the economic analysis of bioethanol production from the plant were carried out by a team of researchers from the University of Sydney (Australia; [www.sydney.edu.au](http://www.sydney.edu.au)), University of Adelaide (Australia; [www.adelaide.edu.au](http://www.adelaide.edu.au)), and University of Exeter (Exeter, U.K.; [www.exeter.ac.uk](http://www.exeter.ac.uk)), led by University of Sydney agronomist associate professor Daniel Tan.

The team says the plant can grow in semi-arid areas without irrigation. It does not

compete with food crops or place demands on limited water and fertilizer supplies. It is heat- and drought-tolerant and can survive Australia's summers.

The team's analysis shows a yield of 7,414 L of bioethanol per hectare per year is achievable with five-year-old agave plants. Although this is lower than that achieved with sugarcane (9,900 L/ha/yr), *Agave* outperforms sugarcane in several ways, including in freshwater eutrophication, marine ecotoxicity, and — crucially — in water consumption. According to the researchers, agave uses 69% less water than sugarcane and 46% less water than corn for the same yield. For U.S. corn ethanol, the yield was lower than that for agave, at 3,800 L/ha/yr. “This shows agave is an economic and environmental winner for biofuel production in the years to come,” says Tan.

(Continues on p. 11)



## Commercial-scale deployment of sodium-ion battery technology

Although Li-ion batteries are currently the dominant technology in the rechargeable-battery sector, sodium-ion (Na-ion) batteries possess many benefits in terms of sustainability, safety and costs, and are poised to greatly expand their commercial usage. Na-ion battery specialist Faradion Ltd. (Sheffield, U.K.; [www.faradion.co.uk](http://www.faradion.co.uk)) — in its largest commercial deployment ever — will provide Na-ion batteries for energy-storage applications in Australia.

Previously, the company had undertaken several prototype demonstrations, including electric bikes, golf carts and small-scale domestic storage applications, but the project in Australia represents a larger magnitude for Faradion's Na-ion technology, explains Jerry Barker, chief technology officer and co-founder of Faradion.

"Our cell chemistry is based on a proprietary Na-layered oxide

cathode. This chemistry provides a world-leading cell specific energy of 150–160 Wh/kg," says Barker, adding that the company expects to generate cells with greater than 190 Wh/kg specific energy within the next 9 months. Another benefit of Faradion's batteries is that they can be produced on existing Li-ion manufacturing lines. Faradion has worked closely with Haldor Topsoe A/S (Lyngby, Denmark; [www.topsoe.com](http://www.topsoe.com)) in scaling up its proprietary cathode material. With comparable cycling and temperature performance, Na-ion batteries also provide improved thermal stability and safety when compared to conventional Li-ion batteries — Faradion has patented a method for the transportation and storage of Na-ion cells wherein cells can be transported without the risk of overheating or exploding. Faradion's batteries are anticipated to enter the Australian market later this year. ■

sults of the initial trials, Krajete GmbH now plans to build a larger system that can filter 25,000 m<sup>3</sup>/h of air. The system will be installed shortly, and the findings from its operation will feed into further development.

### METALS FROM LOW-GRADE ORES

Critical and economically significant metals, such as cobalt and nickel, can be recovered profitably from metal-refining wastes and ores with lower-than-usual concentrations. This was demonstrated by an E.U. project coordinated by VTT Technical Research Center of Finland (Espoo; [www.vttresearch.com](http://www.vttresearch.com)), which developed new, and combined existing, recovery methods. The recently completed, four-year MetGrow+ project had a budget of €7.9 million, and participants from 19 companies, research organizations and universities from nine E.U. countries.

Various stakeholders interested in using low-grade ores and waste can evaluate the valorization of raw-material streams with the webtool developed in the project. The MetGrow Calculator aims to support decision-making by mines, mining-technology developers, recyclers and others. The user can enter, for example, local laterite ore or jarosite from the zinc industry as a raw material and assess the options for utilizing the raw material stream at local waste and energy prices.

In the project, ways were found to increase Europe's self-sufficiency as far as cobalt, nickel and zinc are concerned, for example, by 10–20% using just the material streams studied in the project. The developed methods can additionally be used to recover metals from other material streams. □

## LINEUP

ARKEMA
BADGER LICENSING
ERAMET
EVONIK
GELEST
HONEYWELL
HYOSUNG
INDORAMA
INEOS
INEOS STYROLUTION
INOVYN
KONICA MINOLTA
LG CHEM
LINDE
MERCK
MITSUBISHI CHEMICAL
MITSUI CHEMICALS
PKN ORLEN
SAINT-GOBAIN
SINOPEC
SYNTHOMER
TATA CHEMICALS
TRINSEO
TRONOX

## COVID-19 Response

### Ineos Styrolution donates ABS for safety-glasses production

May 13, 2020 — Ineos Styrolution (Frankfurt, Germany; [www.ineos-styrolution.com](http://www.ineos-styrolution.com)) has donated over 1,300 kg of acrylonitrile butadiene styrene (ABS) to Friedrich-Alexander-University Erlangen-Nuremberg to help produce protective glasses for frontline healthcare workers.

### Sinopec starts up 16<sup>th</sup> fabric line supporting face-mask production

May 9, 2020 — China Petrochemical Corp. (Sinopec; Beijing, China; [www.sinopecgroup.com](http://www.sinopecgroup.com)) has completed the construction of 16 production lines for meltblown non-woven fabric to make materials for face masks. The company's production capacity for these materials now exceeds 13,500 metric tons per year (m.t./yr), enough to make over 13 billion medical face masks.

### Arkema manufactures hand sanitizer in New York

May 7, 2020 — Arkema S.A. (Colombes, France; [www.arkema.com](http://www.arkema.com)) has repurposed a production line at its plant in Geneseo, N.Y. to manufacture 38,000 L of hand-sanitizer solution for donation across several U.S. states.

### Tata Chemicals produces over 1 million L of disinfectant for COVID-19 response

May 7, 2020 — Tata Chemicals Ltd. (Mumbai, India; [www.tatachemicals.com](http://www.tatachemicals.com)) has transformed its chemical plants in Akola and Ankleshwar, India to produce 75,300 L of hand sanitizer. Additionally, the company has manufactured and supplied more than 1 million L of disinfectant.

### Ineos constructs two new hand sanitizer plants in the U.S.

May 4, 2020 — Ineos (London, U.K.; [www.ineos.com](http://www.ineos.com)) has built two new hand sanitizer plants in the U.S. to provide hand sanitizer to hospitals for free. The facilities will be located in Jacksonville, Ark. and Neville Island, Pa. Each site will produce 1 million bottles of hand sanitizer each month to help with the nationwide shortage.

### Indorama begins production of hand sanitizer in the U.S.

April 30, 2020 — Indorama Ventures Public Co. Ltd. (IVL; Bangkok, Thailand; [www.indoramaventures.com](http://www.indoramaventures.com)) adapted its facilities in the U.S. to manufacture more than 50 tons of hand sanitizer.

### Honeywell manufactures hand sanitizer in the U.S. and Germany

April 29, 2020 — Honeywell (Charlotte, N.C.; [www.honeywell.com](http://www.honeywell.com)) has temporarily shifted

manufacturing operations at its chemical plants in Muskegon, Mich. and Seelze, Germany to produce and donate hand sanitizer to government agencies in response to shortages created by the COVID-19 pandemic.

### Saint-Gobain expands production of respirator membrane components

April 29, 2020 — Saint-Gobain S.A. (Courbevoie, France; [www.saint-gobain.com](http://www.saint-gobain.com)) has significantly increased production capacity for silicone membranes that are embedded into respirators. By adapting some of its sites in France, Saint-Gobain has been able to multiply silicone-membrane production by a factor of 15.

## Plant Watch

### PKN Orlen to expand IPA production using Badger process technology

May 13, 2020 — As part of a program to expand phenol production capacities, PKN Orlen (Plock, Poland; [www.orlden.pl](http://www.orlden.pl)) purchased a license and basic-engineering design package for an isopropyl alcohol (IPA) production unit from Badger Licensing, LLC (Boston, Mass; [www.badgerlicensing.com](http://www.badgerlicensing.com)). If the project moves forward, PKN Orlen will be the world's fifth company to implement this IPA technology.

### Evonik commissions biomaterials production facility in Birmingham

May 13, 2020 — Evonik Industries AG (Essen, Germany; [www.evonik.com](http://www.evonik.com)) commissioned a biomaterials manufacturing facility in Birmingham, Ala., which will support increasing global demand for bioresorbable polymers. Together with an adjoining CDMO facility in Birmingham, Evonik can now provide an integrated range of biomaterial services at a single U.S. site.

### New power-to-methanol project announced by Inovyn

May 7, 2020 — Inovyn (London, U.K.; [www.inovyn.com](http://www.inovyn.com)) announced a power-to-methanol project in Antwerp, Belgium, which will produce methanol from captured CO<sub>2</sub> combined with hydrogen generated from renewable electricity. Inovyn will contribute to a joint feasibility study evaluating the construction of an industrial-scale demonstration unit at Inovyn's Lillo manufacturing complex. The planned unit would produce 8,000 m.t./yr of methanol.

### Linde and Hyosung sign MOU for world's largest liquid-hydrogen plant

April 28, 2020 — Hyosung Corp. (Seoul, South Korea; [www.hyosung.com](http://www.hyosung.com)) and Linde plc (Guildford, U.K.; [www.linde.com](http://www.linde.com)) plan to invest in the construction of a liquid hydrogen plant at Hyosung's Yongyeon plant in Ulsan. With production capacity of 13,000 m.t./yr of liquid hydrogen, it will be the world's largest



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liquid-hydrogen manufacturing facility. The companies plan to break ground in the first quarter of 2021 and complete the plant in 2022.

#### **LG Chem to expand carbon nanotube production capacity**

April 28, 2020 — LG Chem Ltd. (Seoul, South Korea; [www.lgchem.com](http://www.lgchem.com)) is expanding its production of carbon nanotubes (CNTs) by 1,200 m.t./yr at the company's Yeosu plant. After the expansion, LG Chem will have a total CNT production capacity of 1,700 m.t./yr.

#### **Mitsui Chemicals to construct new plant for cyclic olefin copolymers**

April 22, 2020 — Mitsui Chemicals, Inc. (MCI; Tokyo, Japan; [www.jp.mitsuichemicals.com](http://www.jp.mitsuichemicals.com)) will establish a new plant in Osaka to produce cyclic olefin copolymers. This plant will increase MCI's production of cyclic olefin copolymers by approximately 50%. Construction on the new plant is slated to wrap up in March 2022.

### **Mergers & Acquisitions**

#### **Tronox acquires Eramet titanium and iron assets in Norway**

May 14, 2020 — Tronox Holdings plc (Stamford, Conn.; [www.tronox.com](http://www.tronox.com)) announced that it has signed a definitive agreement to acquire the TiZir Titanium and Iron (TTI) business from Eramet S.A. (Paris, France; [www.eramet.com](http://www.eramet.com)) for approximately \$300 million. TiZir's TTI facility, located in Tyssedal, Norway, upgrades ilmenite to produce 230,000 m.t./yr of high-grade titanium slag and 90,000 m.t./yr of high-purity pig iron.

#### **Trinseo acquires Synthomer's vinyl pyridine latex business**

May 4, 2020 — Trinseo (Berwyn, Pa.; [www.trinseo.com](http://www.trinseo.com)) completed the acquisition of the vinyl pyridine latex (VP latex) business from Synthomer plc (London, U.K.; [www.synthomer.com](http://www.synthomer.com)). As part of the transaction, Trinseo has established agreements with Synthomer for contract manufacturing the products at Synthomer's production facility at Marl, Germany, where the products will continue to be produced.

#### **Mitsubishi Chemical to acquire all shares of Gelest**

May 4, 2020 — Mitsubishi Chemical Corp. (Tokyo, Japan; [www.m-chemical.co.jp](http://www.m-chemical.co.jp)) has entered into a definitive agreement to acquire all shares of Gelest, Inc. (Morrisville, Pa.; [www.gelest.com](http://www.gelest.com)). Gelest is a manufacturer and supplier of silicones, organosilanes, metal organics and specialty monomers for several end markets, including medical devices, life sciences and microelectronics.

#### **Merck acquires OLED assets from Konica Minolta**

April 24, 2020 — Merck KGaA (Darmstadt, Germany; [www.merck.com](http://www.merck.com)) has acquired the organic light-emitting diode (OLED) patent portfolio from Konica Minolta Inc. (Tokyo, Japan; [www.konicaminolta.com](http://www.konicaminolta.com)). The acquired portfolio comprises over 700 patent families. ■

*Mary Page Bailey*



# Adaptability Helps Flow Instrumentation Evolve

A common adage says “if you can’t measure it, you can’t control it.” Now, advances in flow-measurement technologies are increasing the breadth of what it is possible to measure

## IN BRIEF

ENVIRONMENTAL  
MANAGEMENT

LESS PROCESS  
DISRUPTION

MULTIFACETED FLOW  
DEVICES

As process demands evolve and organizations increasingly focus on sustainability in their operations, chemical processors must stay abreast of what these changes mean for their instrumentation devices, especially those that measure, monitor and control flow. Beyond measuring flowrate, modern flowmeters increasingly must adapt to rapidly changing conditions, measure many parameters beyond flowrate and continuously communicate with other systems in a plant.

### Environmental management

“Customers want more than just a flowmeter, they want solutions. This may be additional hardware or software that allows the flowmeter to interface with another control system in the plant,” says Scott Rouse, director of product management for Sierra Instruments, Inc. (Monterey, Calif.; [www.sierrainstruments.com](http://www.sierrainstruments.com)). Combining adaptability with connectivity, Sierra has developed a mass flowmeter that can be updated with changing gas compositions in real time, integrating with a gas chromatograph to obtain gas composition (Figure 1), explains Rouse. Using Sierra’s qMix gas-mixing application (app), users can create unlimited custom gases or gas mixtures to compensate for gas compositional changes in the field. Measurement of combustion, flaring and waste-gas processes are often regulatory requirements, and Sierra has now developed an advanced qMix functionality, qMix RealTime, for realtime monitoring of flare and stack gases, says Rouse, thus helping users meet the requirements of organizations like the

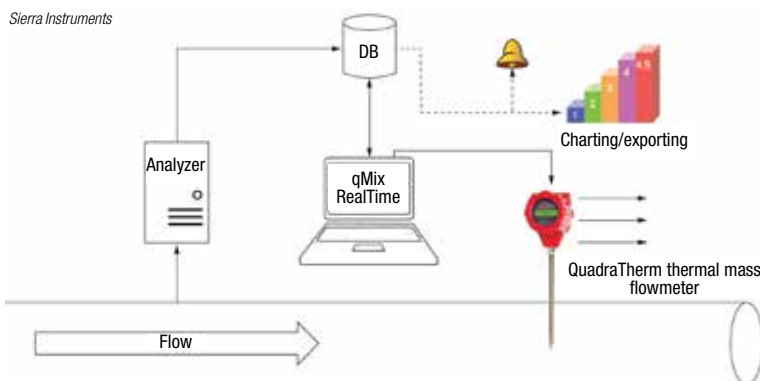


FIGURE 1. The qMix tool integrates with a gas chromatograph to provide realtime measurement of varying gas compositions

U.S. Environmental Protection Agency (EPA) and Bureau of Land Management (BLM).

Changing gas compositions can certainly wreak havoc on flow-measurement accuracy in a wide range of industrial applications, including those processing biogas and natural gas. “In biogas and natural gas applications, gas composition can change and look very different from week to week,” explains Ria Edens, industrial marketing communications manager at Fox Thermal Inc. (Marina, Calif.; [www.foxthermal.com](http://www.foxthermal.com)). Fox Thermal offers the Gas-SelectX tool on several of its thermal mass flowmeters, which allows users to quickly re-program the meter to begin measuring new gas compositions identified by gas sampling. This tool utilizes a library of common pure gases or can be programmed for the users’ own custom gas mixtures. In shale-gas applications, accurate flow measurement of changing gas mixtures is critical for allocation and royalty purposes among the many stakeholders.

Compressed air is another area where accurate flow measurement can reduce wasted energy, thus greatly contributing to efficiency, as problems with leakage are nearly universal to these types of systems. Thermal mass flowmeters provide accuracy benefits with their simplicity — unlike other flow-measurement techniques, they do not require accompanying

pressure or temperature calculations to read flowrate. “What ends up happening is that you have three or more different instruments taking these separate measurements, and each one has its own accuracy, so that compounds the measurement uncertainty you’re dealing with,” explains Edens.

In compressed-air systems, many interconnected sub-meters may be involved, so minimizing uncertainty is key. “You have to figure out where the leaks are happening, so installing a flowmeter to measure primary airflow from the compressor, along with sub-meters at each end-use facility, can give data to compare to the estimated demand. If there is variance, you can start to identify leaks,” she adds.

Wastewater treatment is an essential part of environmental management, and flow measurement is a central activity in ensuring that these processes are operating as intended. Although most users might envision a flowmeter being installed on a closed pipeline or tube, the open channels encountered in the water-treatment sector also require accurate and precise flow measurements, particularly at the water inlet (Figure 2). For these special tasks, devices typically used for level measurement can be adapted. “You can find open channels in all kinds of wastewater treatment systems. The flow is measured in open channels using an ultrasonic sensor for height measurement and related linearization,” explains Jürgen Skowaisa, product manager Radar, VEGA Grieshaber KG (Schiltach, Germany; [www.vega.com](http://www.vega.com)). For this type of application, radar technology provides reliability because it is

unaffected by temperature changes, says Skowaisa. VEGA’s radar devices are now offered with a new 80-GHz radar chip that is optimized for measurement tasks in the water and wastewater industries.

### Less process disruption

Flowmeters are also adapting to processing demands by offering simpler installation and maintenance for their

devices. “Customers are tending to shy away from devices with many moving parts that could cause downtime,” says Dave Vollaie, instrumentation product manager at GF Piping Systems (GF; Irvine, Calif.; [www.gfps.com](http://www.gfps.com)). GF has launched the Signet 2580 FlowtraMag magnetic flowmeter (Figure 3), which features a full-bore, all-plastic design with no moving parts that is lighter weight



**FIGURE 2.** A level-measurement device can be adapted to provide open-channel flow-measurement readings in water-treatment applications



**FIGURE 3.** With no moving parts and less restrictive installation requirements, the Signet 2580 FlowtraMag is suitable for tight spaces and short pipe runs than conventional full-bore magnetic flowmeters.

According to Voltaire, plastic construction provides environmental benefits in that there are no coated metal parts that could chip and cause corrosion or damage within a process. Streamlined installation is also beneficial. "The shorter run requirements after flow disturbances, such as elbows, valves or pumps, enable users to fit these flowmeters into tighter spaces that otherwise would not be possible with other flow technologies," according to Voltaire. Furthermore, adaptive flow measurement in both the forward and reverse direction is possible when Signet meters are used alongside the company's S3L sensors.

Another trend in flowmeter design aims to minimize process disruptions during installation. "We are increasingly observing a trend towards non-invasive and clamp-on instrumentation, both with respect to ultrasonic flow measurement and other analytical applications," says Jörg Wylamrzy, director of sales at Flexim GmbH (Berlin, Germany; [www.flexim.com](http://www.flexim.com)). Last year, Flexim released the world's first non-invasive technology for steam measurement, the Fluxus G601 ST (Figure 4).

Measuring and monitoring steam flow is an essential task to ensure energy-efficient operations and reduce waste. "In particular, non-invasive flow measurement in steam-distribution networks can often reveal a huge energy-efficiency potential for plants," adds Wylamrzy.

Another common application for clamp-on ultrasonic measurement technology is controlling the efficiency of heat exchangers for predictive maintenance tasks. "In general, non-invasive measurement of volume and mass flow, as



**FIGURE 4.** The Fluxus G601 ST is said to be the first non-invasive flow-measurement technology for steam systems

well as concentration and density, allows for increases in energy efficiency without affecting plant availability," continues Wylamrzy. Later this year, Flexim plans to release a new high-temperature steam flowmeter that overcomes challenges associated with high-temperature steam operations.

### Multifaceted flow devices

As flowmeters evolve to handle a wider range of processing demands, users increasingly need multifunctional instruments. "Most flowmeters are starting to be recognized as multivariable devices, providing flow information, but also other process variables, such as density, temperature and viscosity," says Howard Siew, chemical industry manager at Endress+Hauser (Greenwood, Ind.; [www.us.endress.com](http://www.us.endress.com)). "Users are continuing to look for qualitative measurements, not just quantitative ones. They typically use laboratory measurements to verify product quality, but this decreases efficiency and throughput. Through the use multivariable flowmeters, users can monitor live product quality and adjust their process on the fly, if necessary," adds Siew. "Rather than thinking of these devices as flowmeters, it's a change to thinking of them as process analyzers," emphasizes Nathan Hedrick, Endress+Hauser's national flow product manager.

As an example, Adam Booth, flow product marketing manager at Endress+Hauser, highlights the company's recently launched Prosonic Flow G 300/500 inline ultrasonic flowmeter: "This device is available with optional integrated pressure and temperature measurement, which provides an accurate measurement,

even with changing process conditions. It also features the Advanced Gas Analysis application package, which enables the device to compensate for varying gas compositions and provides more process variables, such as energy values, molar mass and density."

To illustrate the importance of compensating for varying process compositions and measuring values beyond flow, Hedrick cites the example of a boiler. "You have some energy stream in (typically natural gas) that is converted to a different energy stream out (steam). With devices like we've mentioned here, you can immediately see the efficiency of the boiler in converting from one energy stream to another by monitoring the associated energy values." One particularly valuable parameter to measure is steam quality — Endress+Hauser's Prowirl 200 vortex flowmeter (Figure 5) can provide a steam quality measurement down to a dryness fraction of 80%. Hedrick explains: "Saturated steam that is 100% dry is essentially a theoretical fluid. As soon as you have any inefficiency in your steam system from the boiler, you start to get condensation. Most flow-measurement devices assume 100% dry saturated steam in any energy calculations they provide. As a result, unless you are using a device with wet-steam measurement, or a separate steam-quality-measuring device, you are getting an overinflated energy value from the device," he says.

In hygienic or sanitary processes, advanced multiparameter meters provide many benefits — not only for producing high-quality products, but also for controlling the frequent rinsing and



**FIGURE 5.** The Prowirl 200 vortex flowmeter provides information on steam quality alongside conventional flowrate measurements



cleaning activities that are required to ensure process integrity. “Many modern flow-measurement devices are multiparameter sensors. Besides the flowrate, they are able to measure other process-relevant parameters in a single device,” says Hendrik Faustmann, product manager, FLOWave at Bürkert Fluid Control Systems (Ingelfingen, Germany; [www.burkert.com](http://www.burkert.com)).

In the case of Bürkert’s FLOWave flowmeter (Figure 6), surface acoustic wave (SAW) technology is employed to obtain a direct measurement of flow, combined with temperature readings, interpretations for density and an acoustic transmission factor. This factor provides an indication of viscosity, as well as the presence of solids or bubbles in the measured fluid. “Together, these parameters can be analyzed to provide real-time analysis of the contents of a pipeline, for instance to detect media changeover in rinsing or clean-in-place processes,” explains Faustmann. This enables the flowmeter to rapidly adapt to changeovers between different liquid types during production. “Particularly in cleaning processes, rapid differentiation between product and rinsing water or cleaning reagents ensures efficient process control. Compared to conventional time-controlled processes, product waste can be minimized,” adds Faustmann. These meters do not require that sensors come into contact with process media, and the measurements are taken independently of conductivity, making them suitable for hygienic processes, such as those in pharmaceutical manufacturing.

With the many available flowmeter technologies, it can be difficult

*Bürkert Fluid Control Systems*



**FIGURE 6.** The multiparameter readings provided by FLOWave devices give many valuable parameters for controlling clean-in-place operations in addition to regular production processes

for users to select just one device to best suit their processing needs. Fluid Components International LLC (FCI; San Marcos, Calif.; [www.fluidcomponents.com](http://www.fluidcomponents.com)) has launched the Adaptive Sensing Technology (AST) for its ST80 flowmeters (Figure 7), which brings together the two major modes of operation for thermal-mass gas-flow measurement — constant power (CP) and constant temperature (CT) — said to be an industry-first achievement.

“CT-type devices historically give a faster response but have less range, and use more power” explains Randy Brown, executive director of marketing and industrial sales & service for FCI. “On the other hand, CP devices can have slower response, but provide more range and consume less power,” he continues. FCI developed and patented the AST sensor drive technology, which enables its flowmeters to adaptively shift between the two modes, depending on the measured value. “The instrument starts out in CT mode, and as the flowrate increases, it will switch automatically over to CP,” says Brown. This adaptability provides users with the fast response of CT meters combined with the wider flow range and lower power needs of CP meters.

Further, since standard CT devices react more erratically when there is moisture or humidity in the pipe, the ST80 can be fixed to operate only in the CP mode to deliver a stable reading. This type of adaptability is particularly beneficial in new process applications, where users may not always know the specific conditions inside of a pipe, says Brown. “In the field, users can switch the unit’s operating mode from AST to CP or CP to AST, to provide the optimum performance in an installation’s actual conditions as opposed to replacing it with a different device.”

While flow measurement is the paramount function, many other variables beyond a process’ flowrate must be considered to ensure flowmeters’ adaptability and reliability. Emerson Automation Solutions (Austin, Tex.; [www.emerson.com](http://www.emerson.com)) has developed several new tools aimed at providing more insight

*FCI*



**FIGURE 7.** The new AST sensor head enables hybrid operation for thermal mass flowmeters, enabling measurement accuracy across a wider flow range

into the flow-measurement process to support improved data use and instrument diagnostics.

For instance, the Smart Meter Verification diagnostic suite allows users to run a health check for an in-service device, looking at many factors that can help streamline troubleshooting and maintenance. “There is a version designed for both magnetic and Coriolis flowmeters that helps extend operating intervals by detecting things like non-uniform coating, erosion and corrosion,” explains Tonya Wyatt, product line manager at Emerson Automation Solutions. Another tool, Zero Verification, helps users with flowmeter calibration. “This tool is especially helpful where the mass flowrate is on the low end of the meter range in applications such as low-pressure gases, high-viscosity fluids and other applications with a large turndown ratio,” mentions Wyatt.

Emerson also offers Advanced Phase Measurement, a patented set of algorithms used on Coriolis meters to improve the accuracy of flow measurement in multiphase flow conditions, including liquids with entrained gas and wet gases. It can also be used in concentration measurements when gas is present, including in oil, water or gaseous mixtures. With so much data available, it is important that users not be overwhelmed. Emerson’s ProcessViz visualization software, says Wyatt, helps people get a quick view of all the data being created by the instrumentation without having to spend hours manipulating data in large spreadsheets.

*Mary Page Bailey*

# Focus on Milling

Gericke USA

## Milling systems adjust at top for safety and efficiency



Van Tongeren America

This company recently unveiled a line of cone mills (photo) for fine-particle size reduction. Developed for milling materials from 150  $\mu\text{m}$  to 12 mm, these cone mills feature a patented design that allows the gap between the rotor and screen to be quickly adjusted based on the targeted particle size and shape from outside the machine without requiring any disassembly or contact with the cutting chamber. With a built-in gap-adjustment indicator verifying the selected particle size, efficiency, versatility and worker safety are achieved. The cone mills move dry, moist, fatty, sticky and heat-sensitive materials over a conical, serrated screen, passing on-spec particles through to discharge and recirculating larger particles back to the cutting chamber for further size reduction. Creating a high-impact cutting action at a low velocity, the cone mills quickly advance the material through the grinding zone with minimal heat, dust or noise generation. Available in three standard models, the cone milling equipment includes a quick-release access to the cutting chamber and rotor assembly for safe, easy cleaning and screen changes, plus food-quality seals and a choice of screen sizes, hole shapes and patterns. Custom-engineered models and a variety of custom options are available. — *Gericke USA, Inc., Somerset, N.J.*

[www.gerickegroup.com](http://www.gerickegroup.com)

## A classifier for separating dry particles

This company has launched a Centrifugal Classifier (CC; photo) that harnesses air, gravity and centrifugal force to automatically separate dry particles without any water. Featuring a proprietary design, the pneumatic-air classifier separates the feed material from the airstream and redirects the air into a curtain of material to create a scrubbing action that directs larger particles to the discharge outlet and directs fine particles into a controlled vortex en route to dis-

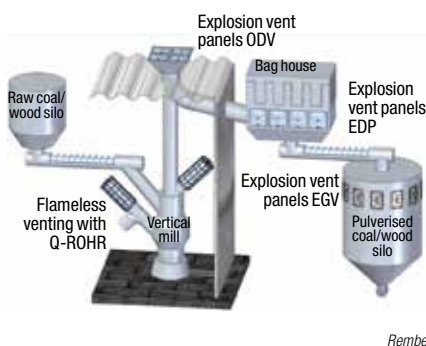
charge with the airstream to a dust collector. Designed with no moving parts, the CC offers an efficient alternative to screening machines with virtually no maintenance, no work stoppages for screen cleaning and replacement, and at lower initial and operating costs. Developed for classifying cement, alumina, salt, potash, limestone, fertilizer and other materials, the CC separates dry particles at any cut point between 100 to 15  $\mu\text{m}$ . The classification system is custom-designed and manufactured for each installation to meet target production goals and material specifications, and is proven to perform continuously in unattended, continuous operation. — *Van Tongeren America, LLC, Lebanon, Pa.*

[www.van-tongeren.com](http://www.van-tongeren.com)

## Explosion protect the milling area of a co-firing power plant

This company has developed a new safety approach for protecting coal or wood grinding plants against the ravages of a combustible-dust explosion. Through the combination of standard explosion panels and flameless venting, protection can be guaranteed. The system (photo) is composed of the following components: the flameless venting technology, which enables indoor venting in the rolling-mill building; to protect the baghouse and isolate the inlet ductwork from a combustible dust explosion, standards panels, such as the company's ODV and EDP, can be utilized; finally, the storage silo can also be protected by standard vent panels. In the case of silos, there is little to no vacuum pressure in a silo so an EGV device can properly protect this enclosure. Isolation protection is also required to stop the propagation of any combustible dust explosion — this can be accomplished through numerous passive approaches. Also, if the filter and silo happen to be located indoors, then they can also be protected by flameless venting devices. — *Rembe Inc., Charlotte, N.C.*

[www.rembe.us](http://www.rembe.us)



Rembe

### Hammer mills for reducing soft to medium-hard materials

The ideal hammer mill is determined by feed size, feed rate, operating conditions and desired product output. This company offers many configurations of industrial hammer mill grinders (photo) that transform the consistency of soft and rough materials by using the pulverizing technique of a hammer mill grinder to produce a finished particle. Capacities from 1 to 300 ton/h are offered with 30- to 250-hp models. Capacities vary depending on feed size, feedrate, operating conditions, desired product output, characteristics of feed material, and equipment configuration. Perforated screens or grate bars are used for reducing soft to medium hard materials with multiple size-reduction stages. Fibrous materials are easily handled due to the high concentration of shearing actions by the grate bars or screen within the unit. Specially designed heavy reversible hammers, working in conjunction with the company's adjustable breaker plate, enables the hammer mill to produce a uniform product with the lowest possible maintenance. — *Stedman Machine Co., Aurora, Ind.*

[www.stedman-machine.com](http://www.stedman-machine.com)

### High-capacity cone mill for sticky, moist fragile materials

A new 29.5-in.-dia. (750 mm) cone mill, CGM 750 (photo), gently grinds fatty, heat-sensitive, sticky, moist or fragile materials into uniform particle sizes with minimal fines at rates up to 35 ton/h (32 m.t./h). Vertical material flow and low-energy grinding action with short residence times yield close particle-size distribution from 125 to 250 microns, without the noise, dust, product build-up or heat generation associated with traditional mills, says the manufacturer. Ideal for bulk pharmaceuticals, foods and contamination-sensitive chemicals, the sanitary unit is suitable for wet and dry milling, pre-conditioning and de-agglomerating of a wide range of bulk products. Material is fed by gravity or vacuum-conveying system into a central milling chamber, in which a rotating impeller accelerates it centrifugally against and through apertures in a conical screen, and then into a re-

ceptacle below the milling chamber. A pneumatic, swing-down conical discharge chute with quick-release clamps allows easy lowering of the 250-lb (114-kg) cone housing and full access to the housing interior, beater and perforated conical screen for cleaning and sanitizing. The unit can be cleaned wet or dry, and is available configured for clean in place (CIP), steam in place (SIP) or filling with liquid during cleaning. Provision is included for optional air purging of bearings and seals. Conical screens with round, square or raised-lip (cheese grater) perforations can be changed rapidly and are offered with apertures from 150 micron for fine/dry de-agglomeration, and up to 25 mm for wet granulation. Constructed of 304 or 316 stainless steel to sanitary or industrial standards, the mill is top-driven by a belt drive and equipped with fully interlocked moving components with time-delay function for operator safety. Explosion-containment versions are also available. — *Kason Corp., Millburn, N.J.*

[www.kason.com](http://www.kason.com)

### Prepare reliable, representative samples in a very short time

The Knife Mill Pulverisette 11 (photo) produces a homogeneous sample in a very short time due to its special design. Each subsample taken from any location in the grinding vessel is representative for the original sample and thus ensures an exact, significant analysis; this holds for a wide range of different materials, regardless of whether the sample is dry, moist, soft, medium-hard, fibrous or oily. The system is ideal for sample preparation in the fields of foodstuffs or animal feed testing, agriculture and forestry, biotechnology, pharmaceuticals and chemicals. The standard knife is made of stainless steel and can be used for comminution of nearly all materials, due to its four blades and geometry. For comminution of brittle, hard samples, a sickle knife made of stainless steel can be used. The sickle knife with serrated blades is ideal for grinding especially fibrous, tough or sinewy samples, such as fish, meat, asparagus or bamboo. — *Fritsch GmbH, Idar-Oberstein, Germany*

[www.fritsch.de](http://www.fritsch.de)

Gerald Ondrey

Stedman Machine Company



Kason



Fritsch



# New Products

Labom Mess- und Regeltechnik



Endress + Hauser

## This DP transmitter operates over a wide temperature range

The new Pascal CI4350 differential-pressure (DP) transmitter (photo) is equipped with filling-level software and can display differential and static pressure at the same time. The device has a high-definition graphic display, which makes comprehensive configuration possible, as well as simulations and diagnostics. The CI4350 is particularly suitable for very high process temperatures with a maximum operating pressure of up to 160 bars as standard (up to 400 bars as an option). The Pascal CI4350 is not only suitable for high process temperatures (the measuring range is  $-90$  to  $400^{\circ}\text{C}$ ), but is also designed for aggressive media — the wetted parts can optionally be made of stainless steel, tantalum, hastelloy or polytetrafluoroethylene (PTFE).

— Labom Mess- und Regeltechnik GmbH, Hude, Germany

[www.labom.com](http://www.labom.com)

## A new, 80-GHz, wireless radar-level sensor

The FWR30 is this company's first cloud-connected radar level sensor, and is said to be the world's first 80-GHz wireless IIoT (industrial internet of things) sensor, providing full transparency in the storage and transport of liquids. The instrument's continuously recorded measurement data can be accessed at any time, from any location, due to the device's cloud connection, with communication made possible by an integrated SIM card. The compact device is suitable for stackable tanks and offers flexible and easy installation. In addition to the measured level, the instrument provides information on tank location, helping users optimize logistic and storage processes and save time by providing continuous access to inventory information. The free-radiating measuring device covers measuring ranges up to 50 ft and temperatures between  $-4$  and  $140^{\circ}\text{F}$ . Because it uses non-contact measurement, the Micropilot FWR30 can be used in all liquid media. It is resistant to corrosion, abrasion, viscosity and toxicity.

— Endress+Hauser, Greenwood, Ind.

[www.us.endress.com](http://www.us.endress.com)

## Full-scale testing services to optimize screening productivity

Ensuring optimal screening processes and productivity is vital for a vast range of processing needs. This company recently expanded its 10,000-ft<sup>2</sup> Materials Testing Laboratory (photo), which conducts advanced screening testing. The lab's experts recently worked on a full-scale material-recovery test related to the recovery of carbon black and other chemicals from end-of-life tires, with the goal of achieving a specific size of ground-rubber particles. The Material Testing Lab was able to determine the required feed quality for the desired particle size, and also qualified the type of equipment that was necessary to improve consistency. The company offers these full-scale tests free of charge. The testing process may involve a screening questionnaire, material-sample submission, feasibility tests and more. Tests are run on full-sized machinery to simulate real-world conditions with test materials.

— Midwestern Industries, Inc., Massillon, Ohio

[www.midwesternind.com](http://www.midwesternind.com)

## Flow conditioners enhance ease pump installation

The efficiency and service life of pumps can be greatly extended by following the manufacturer's installation recommendation for pipe straight run entering the pump. When cramped pump houses or restricted pipe runs make this impractical and costly, this company offers a range of flow conditioners (photo) to overcome these challenges. The devices deliver a uniform, swirl-free flow profile to the pump inlet in as little as six pipe diameters by correcting flow disturbances to mimic adequate pipe straight run and produce a highly repeatable, symmetrical flow profile. The flow conditioners' anti-swirl and inclined vortex-generating profile-correction tabs, projecting from the inside pipe surface, generate vortices that accelerate these natural pipe effects to create a uniform flow profile. Their use also eliminates the extra pipe cost and technician labor for additional lengths of pipe straight run or moving equipment.

— Vortab Co., San Marcos, Calif.

[www.vortab.com](http://www.vortab.com)



Midwestern Industries



Vortab

### Universal input display has transmitter and alarm features

The new VPM3000 Series of universal input displays (photo) with transmitter and alarm capabilities combines the digital indicator function of a panel meter with optional signal conditioning for 4–20-mA transmitter output and alarm trip solid-state relays. The devices are able to operate as a transmitter or alarm to satisfy a wide variety of process and temperature applications. Field-selectable inputs accept process current/voltage and temperature sensor signals, including 4–20 mA,  $\pm 20$  mA, 0–10 V,  $\pm 10$  V, Pt resistance temperature detectors (RTDs), and most common thermocouple varieties. For additional versatility, units can provide power to drive a 4–20-mA transmitter and other instruments. Modbus RTU serial communication is also supported. — *Acromag, Inc., Wixom, Mich.*

**[www.acromag.com](http://www.acromag.com)**

### High-speed gas analysis enables emissions reductions

The TDLS8100 probe-type tunable diode-laser-spectrometer (TLDS) realtime gas analyzer (photo) makes quick and accurate in situ gas-concentration measurements, eliminating the requirement for sampling and conditioning components in industrial process-heating applications. These capabilities support safety improvements, emissions reductions, optimization of fuel efficiency and yields, as well as prolonging the life of assets. The instrument is field-serviceable with no consumables or calibration required. This results in reduced installation and maintenance costs. According to the manufacturer, the single-flange design allows installation where cross-duct TDLS technology was previously not feasible due to accessibility, obstructions or budget constraints. For use in safety instrumented systems, the TDLS8100 analyzer is compliant with safety integrity level 2 (SIL 2). It is also explosion-proof for Division 1 and Zone 1 installations. — *Yokogawa Corp. of America, Sugar Land, Tex.*

**[www.yokogawa.com/us](http://www.yokogawa.com/us)**

### A next-generation condition-monitoring field device

The next-generation VCM-3 condition-monitoring platform (photo) is designed to increase the uptime and reduce overall lifecycle costs of pumps, fans, motors, gearboxes and many other types of machines, while increasing reliability. The field-proven VCM-3 is a 12-channel data-acquisition hub for monitoring auxiliary machines, balance-of-plant (BOP) non-critical machines and other assets in industries where cost-effective condition monitoring is required without machine protection. It features enhanced condition-monitoring measurement techniques, easy system integration, built-in cybersecurity and simple installation. The platform has been designed for scalability, so users can get started quickly with the basic model. — *Brüel & Kjær Vibro GmbH, Darmstadt, Germany*

**[www.bkvibro.com](http://www.bkvibro.com)**

Acromag



Yokogawa



Brüel & Kjær Vibro



### Two new vacuum pumps for mass spectrometer systems

Hena 50 (photo) and 70 are single-stage, oil-sealed rotary vane pumps that were developed for the demanding requirements of mass spectrometer systems. They achieve pumping speeds of between 32 and 59 m<sup>3</sup>/h, depending on their size and speed of rotation. Their integrated oil-mist separator ensures clean exhaust air. They are equipped with a frequency converter that enables them to be used worldwide with single-phase input and identical pump capacity for 50 and 60 Hz. A constantly high flowrate in the target pressure range, adjustable pumping speeds and low final vacuum contribute to the instrument's reliable high performance. Long maintenance intervals and operating hours are ensured by the high oil volume in the pumps and their low oil temperature during operation. — *Pfeiffer Vacuum GmbH, Asslar, Germany*  
**[www.pfeiffer-vacuum.com](http://www.pfeiffer-vacuum.com)**



Siemens Digital Industries

### Locating software includes digital twin of performance

The new Location Intelligence software (photo) expands the Simatic RTLS (realtime locating system) platform to include a digital twin of performance. The web-based software analyzes and visualizes dynamic data, processes events, and can be addressed directly from enterprise resource planning (ERP) or manufacturing execution system (MES) systems. The position data provides the user with a comprehensive overview of material flow, order information or possible problem areas. Depending on the process step and order status, order information can then be displayed on the Simatic RTLS ePaper transponders, providing a paperless solution. — *Siemens Digital Industries, Nuremberg, Germany*  
**[www.siemens.com](http://www.siemens.com)**



Lauda Dr. R. Wobser

1.2 kW at 20°C. The Semistat S 2400 incorporates the latest innovations from the thermoelectrics sector, including high-performance Peltier elements, which guarantee a higher power density and greater reliability. The Semistat 4400, with 4.4-kW cooling output, is designed for applications using 300-mm wafers with a high cooling-output requirement at low temperatures. — *Lauda Dr. R. Wobser GmbH & Co. KG, Lauda-Königshofen, Germany*  
**[www.lauda.de](http://www.lauda.de)**

### Durable analytical balances provide improved safety features

The MS-TS analytical balance (photo) features a robust full-metal housing, as well as built-in quality-assurance and safety features designed for shared laboratory equipment. In addition to being easy-to-clean, the MS-TS offers Glove Mode, which makes the touchscreen even more sensitive and easy to navigate through gloves, reducing the risk of exposure to aggressive materials. Multiple interface options make it easy to manage data, and with the addition of EasyDirect data-management software, users can collect data from up to 10 balances simultaneously for fast, error-free reporting and enhanced regulatory compliance. — *Mettler Toledo GmbH, Greifensee, Switzerland*  
**[www.mt.com](http://www.mt.com)**



Mettler Toledo

### This pilot-scale mixer has been redesigned

The newly redesigned RS-02 high-shear pilot-scale mixer (photo) is well suited for pilot plants and small-scale production, including product development, formula optimization and trial mixtures. The RS-02 redesign includes a mobile lift stand with a wider base for added stability and a hand crank for adjusting mixer height. The portable unit fits through standard-width doorways, making it easier to transport it within a plant. The RS-02 has a batch capacity of 5 to 20 gal and incorporates the patented Rotosolver high-shear mixing impeller to provide balance between shear and flow so that mixtures are completely homogenous, emulsified and agglomerate-free. — *Admix, Inc., Londonderry, N.H.*  
**[www.admix.com](http://www.admix.com)**  
*Mary Page Bailey and Gerald Ondrey*



Admix

### Temperature control for the semiconductor industry

The new Semistat series of thermoelectric process thermostats (photo) are used to control temperature in etching processes in the semiconductor industry. Semistat S 1200 is an entry-level device that offers a small footprint with a cooling output of



## Water Treatment: Ion Exchange Resins

Department Editor: Scott Jenkins

Removal of dissolved ions from industrial process water, boiler feedwater or wastewater is a common challenge for facilities in the chemical process industries (CPI). Ion exchange (IX) systems offer one possible solution for demineralization, purification or wastewater-treatment applications. Ion exchange systems facilitate reversible chemical reactions in which the ions targeted for removal are captured electrostatically and replaced by other ions of similar charge. Key components of IX systems are ion exchange resins. This one-page reference provides an overview of how IX resins work in industrial water treatment.

### Resin characteristics

IX resins are generally composed of crosslinked organic polymers shaped into small beads usually measuring between 0.25 and 1.25 mm in diameter. The most common IX resin material is polystyrene with divinylbenzene crosslinks. This type of resin is used in the majority of IX applications. Although the basic resin components are often the same, the resins have been modified in many ways to meet the requirements of specific applications and provide longer resin life.

The polymer matrix of the beads contains dispersed ion-active sites, which are charged functional groups covalently bound to the polymer backbone. The charged functional groups, which can be negative or positive, attract ions of the opposite charge through electrostatic interactions. For example, a common functional group found in cation IX resins is the sulfonate anion ( $\text{SO}_3^-$ ). A counterion solution containing sodium cations ( $\text{Na}^+$ ) is applied to the resin. The  $\text{Na}^+$  ions are held to the bound  $\text{SO}_3^-$  anions by electrostatic attraction, so the resin carries a net neutral charge (Figure 1).

Counterions for the resin are chosen such that when they are exchanged and are released into the treated water, they will not present problems for subsequent use,

and the problematic ions will be removed. In general, the greater the size and valency of an ion, the greater affinity it will have with ions of an opposite charge.

### IX operations

In general, water containing undesirable ions flows through a bed of resin beads and the problematic ions are exchanged for ions of similar charge that were first applied to the resin. The functional groups and initial counterions are designed such that the ions in the treated water will have a greater affinity for the charged functional groups and will dislodge the existing ions and take their place, bonding with the functional groups through shared electrostatic attraction [2].

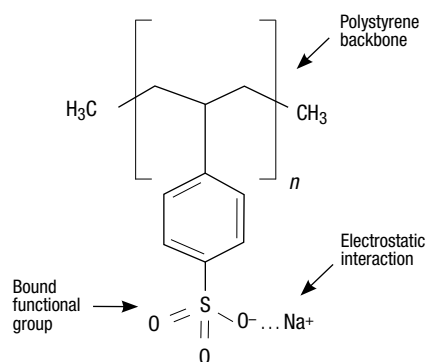
For example, in water-softening applications, water containing  $\text{Ca}^{2+}$  or  $\text{Mg}^{2+}$  ions flows through the resin bed. Since the  $\text{SO}_3^-$  functional groups have a greater affinity for the hardness cations than for the  $\text{Na}^+$  ions, the hardness ions displace the  $\text{Na}^+$  ions, which then flow out of the IX unit as part of the treated stream, while the  $\text{Ca}^{2+}$  or  $\text{Mg}^{2+}$  are retained by the IX resin [2].

The impurities are captured from the feedwater until they saturate the IX resin material, after which they must be flushed out with a regenerant solution. These typically consist of concentrated salts, acids or bases. By flushing the resin, the contaminant ions are released into a waste stream, and the cations and anions in the resin are restored for another cycle.

Resins are designed to resist thermal degradation and oxidizing agents, as well as organic fouling. In addition to polystyrene-divinylbenzene resins, there are newer resins with an acrylic structure, which increases their resistance to organic fouling.

### Resin categories

The ionizable groups attached to the resin bead determine the functional capability of the resin. Generally, in-



**FIGURE 1.** Charged functional groups covalently bound to the polymer backbone of the ion exchange resin are key to its ability to remove undesirable ions from treated water

dustrial-water-treatment resins are classified into four categories:

**Strong acid cation (SAC) exchange resins.** SAC resins are composed of a polystyrene matrix with a sulfonate ( $\text{SO}_3^-$ ) functional group that is either charged with sodium ions for water softening applications, or hydrogen ions ( $\text{H}^+$ ) for demineralization.

**Weak acid cation (WAC) exchange resins.** WAC resins are composed of an acrylic polymer with carboxylic acid functional groups. Due to their high affinity for hydrogen ions ( $\text{H}^+$ ), WAC resins are typically used to selectively remove cations associated with alkalinity.

**Strong base anion (SBA) exchange resins.** SBA resins are typically composed of a polystyrene matrix with fixed anions at exchange sites that yield either chloride ions or hydroxide ions ( $\text{OH}^-$ ).

**Weak base anion (WBA) exchange resins.** WBA resins are unique in that they do not have exchangeable ions, and are therefore used as acid absorbers to remove anions associated with strong mineral acids.

Chelating resins are also available for selective removal of certain metals and other substances.

### References

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2. SAMCO Technologies, "Understanding the Basics of Ion Exchange Systems," SAMCO, e-book, 2017.
3. Dupont Water Technologies, Ion Exchange Resins Tech Fact, Dupont, 2019.

## Production of Orthophthalic Unsaturated Polyester

By Intratec Solutions

Unsaturated polyester resins (UPRs) are solutions of unsaturated polyesters in copolymerizable monomers, which undergo cross-linkage to form thermosetting plastics. The unsaturated polyesters are produced by polycondensation of at least one unsaturated dicarboxylic acid with diols, while styrene is usually employed as a cross-linking agent.

A wide range of UPRs is manufactured according to the intended processing and end-use properties. For example, orthophthalic formulations are used in marine crafts, marbles, gel-coat, buttons; isophthalic resins are used in storage tanks, pipes, pultruded profile and electrical-grade laminates; and dicyclopentadiene formulations are employed in molded electrical components, bathtub products and marine applications.

### The process

The present analysis covers orthophthalic UPR production. The process (Figure 1) comprises three major sections: (1) esterification; (2) cross-linking; and (3) glycols recovery.

**Esterification.** This first step involves the polycondensation between a combination of phthalic anhydride and maleic anhydride, and multiple glycols. Initially, the glycols — monoethylene glycol (MEG) and diethylene glycol (DEG) and propylene glycol — are charged to the reactor. Subsequently, maleic anhydride and phthalic anhydride are added to the mixture. The esterification reactor is

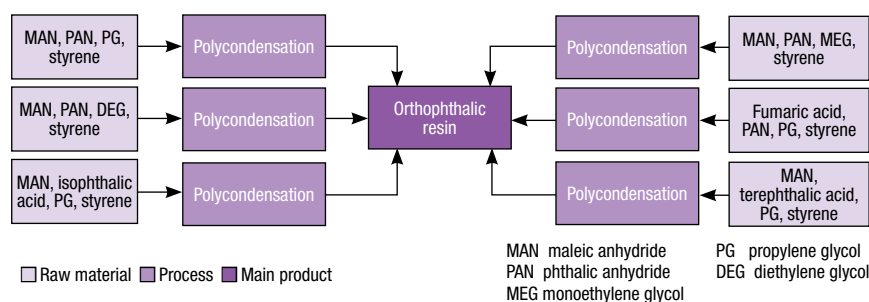


FIGURE 2. Shown here are several production pathways for orthophthalic polyester resin

equipped with stirrers and heating. It is fed with nitrogen to provide an inert blanket. The high temperatures in the reactor cause the evaporation of the glycols and the water generated in the reaction.

**Cross-linking.** Cross-linking is carried out in a different vessel, where the polycondensation product is dissolved in styrene and the reaction takes place. In this reaction, the vinyl radical in the styrene monomer reacts with the unsaturation in the polyester chains, thus linking different chains. Also during this step, promoters are added to initiate the copolymerization reactions, and inhibitors are added to moderate the reaction and keep temperature within required limits to avoid product degradation. At last, the final product is put into drums and stored in a warehouse.

**Glycols recovery.** A water separation column receives the overhead vaporous glycol-water mixture from the reactor. The column top temperature is kept at 100–105°C in such a way that the column overhead product contains only water, which

is further routed to a condenser and discarded. The glycol-rich bottom product is recycled to the reactor.

### Production pathways

Commercial UPR production is mostly based on batchwise condensation of dicarboxylic acids or anhydrides with diols. The specific acids and diols employed may vary according to the product properties need or market price opportunities. Different feedstock formulation examples are presented in Figure 2.

### Economic performance

The total operating cost (raw materials, utilities, fixed costs and depreciation costs) estimated to produce orthophthalic resin was about \$1,200 per ton in the second quarter of 2016. The analysis was based on a plant constructed in the U.S. with capacity to produce 70,000 metric ton per year of orthophthalic resin.

This column is based on “Orthophthalic Unsaturated Polyester Production Process – Cost Analysis,” a report published by Intratec. It can be found at: [www.intratec.us/analysis/unsaturated-polyester-resin-production-cost](http://www.intratec.us/analysis/unsaturated-polyester-resin-production-cost).

Edited by Scott Jenkins

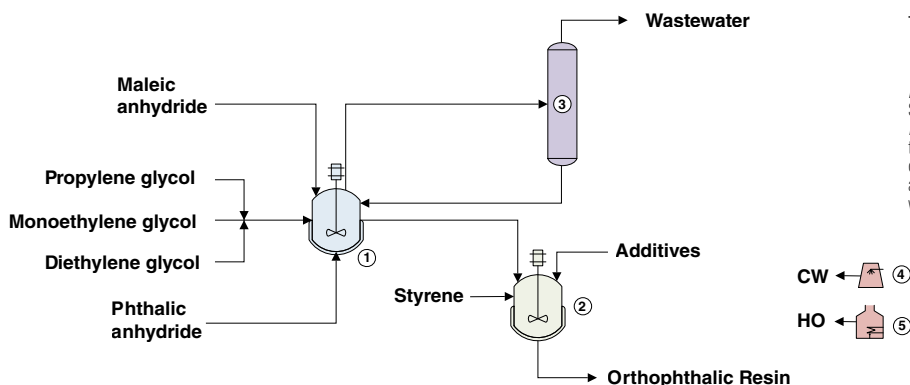


FIGURE 1. The diagram shows a typical batch process for manufacturing orthophthalic polyester resin

**Editor's note:** The content for this column is supplied by Intratec Solutions LLC (Houston; [www.intratec.us](http://www.intratec.us)) and edited by Chemical Engineering. The analyses and models presented are prepared on the basis of publicly available and non-confidential information. The content represents the opinions of Intratec only. More information about the methodology for preparing analysis can be found, along with terms of use, at [www.intratec.us/che](http://www.intratec.us/che).

1. Reactor
  2. Dissolution tank
  3. Water separation column
  4. Cooling tower
  5. Heating oil unit
- HO Heat-transfer oil  
CW Cooling water

# Gas Handling: Process Control for Compressible Flow

Controlling flow in gas-handling applications can present challenges because of the fluids' compressibility. This article presents an overview of how compressible gases behave in response to interacting variables

**Walt Prentice**

Applied Flow  
Technology

## IN BRIEF

COMPRESSIBLE FLOW

PRESSURE AS DRIVING  
FORCE

FLOW MEASUREMENT  
AND CONTROL

PRESSURE CONTROL

CONTROLLING THE  
DRIVING FORCE

Gas-handling operations are common in almost every sector of the chemical process industries (CPI). Some examples are obvious, and others not as much so. Process plants may have heating, ventilation and air conditioning (HVAC) systems for recirculating air; auxiliary steam systems for heating unit operations; or even compressed air for pneumatic valve and tool operation, among others. While controlling parameters such as flowrate and pressure in a gas-handling process may be an intuitive goal, the methods of achieving it may not be. There are certainly physiochemical reasons to control flow and pressure, but there are regulatory reasons as well. For example, controlling pressure drop to keep the sound levels from getting too high in a process plant is a major topic in the Occupational Safety and Health Administration's (OSHA; Washington, D.C.; [www.osha.gov](http://www.osha.gov)) Technical Manual.

### Compressible flow

Process control involving gases has many different aspects, but this article presents a high-level discussion of process control from the fluid system perspective. Chemical engineers need to understand how process fluids behave in response to changing variables. However, the details of process con-

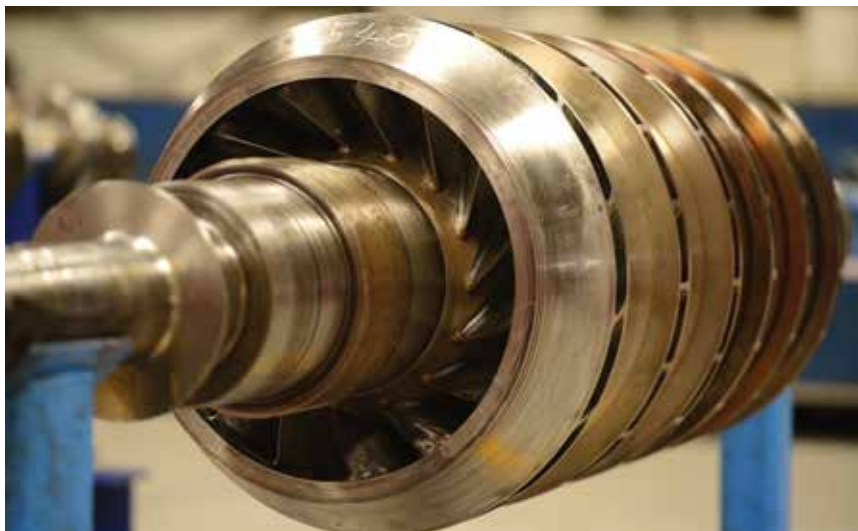


**FIGURE 1.** Since gases are compressible, variable densities alter volumetric flowrate and velocity. This compressible flow presents unique challenges to facilities in the chemical process industries

trol become especially cloudy when working with process gases because of their compressibility (Figure 1).

Compressible flow presents unique challenges and requires special considerations because density is a function of temperature and pressure. This relationship is represented by equations of state, which are often not considered with liquid systems. With gases, this changing density changes volumetric flowrate and velocity. In a theoretical adiabatic (no heat transfer to or from surroundings) pipe of constant cross-sectional area, the fluid velocity increases along with dropping pressure down the line — even though the mass flowrate is steady. In compressor systems, the heat of compression must be considered. This changes the volumetric flowrate — although the mass flow is constant.

Because gas handling operations typically



**FIGURE 2.** Centrifugal compressors can generate different steady-state flowrates with a single motor speed

involve compressible flow, they are complicated by sets of coupled variables, among which are flow and pressure. Gas handling processes require precise control of flow and pressure. To understand the process control for these parameters, you need to understand the relationship between the two.

### Pressure as driving force for flow

The discipline of chemical engineering has a unique focus on transport phenomena. Chemical engineers understand the unifying cornerstone that any transfer of mass, energy and momentum materializes from a driving force. In the case of industrial gas handling, pressure is that driving force for momentum transfer. It helps to remember that pressure is dimensionally consistent with momentum flux. A pressure differential causes a process fluid to spontaneously move from high to low pressure, in accordance with the second law of thermodynamics.

In other words, flow occurs when a difference in pressure is present. When talking about flow and pressure control as separate controlled variables, we might ask “are we really controlling one or the other?” The answer is that the two parameters are not independent of one another; we really control and affect both. It is just a matter of what the objective is, and the reference point.

In operations involving gas han-

dling, there is often a desire to control one parameter over the other: either the goal is a certain flowrate, or a certain pressure. However, there is commonly a secondary goal to keep the counterpart in check — for example, to control the flowrate while ensuring that the pressure does not exceed the maximum allowable operating point. In these situations, it becomes important to remember pressure and flow are not independent of one another.

### Flow measurement and control

First, let’s discuss cases where flow measurement and control are the primary objectives.

It is common to specify flowrates as mass flowrates when discussing compressible flow. The conservation of mass is a fundamental principle, while the conservation of volume is not. However, it is quite common for engineers to measure and handle volumetric flow because of the ease of direct measurement at a single point. In gas-handling systems with changing fluid densities, engineers need to pay careful attention to the type of measurement taken.

Flow readings may come from measuring the pressure drop across an orifice plate, the displacement of a ball flowmeter, or by measuring the velocity with a pitot tube. However, the volumetric flowrate at this single measurement point is not constant down the rest of the pipe. Pressure



and temperature data are required to convert the volumetric measurement into a mass flowrate. Controlling volumetric flowrate may be the end goal, but to do a system analysis, you will need to know the mass flowrate for fundamental continuity analysis.

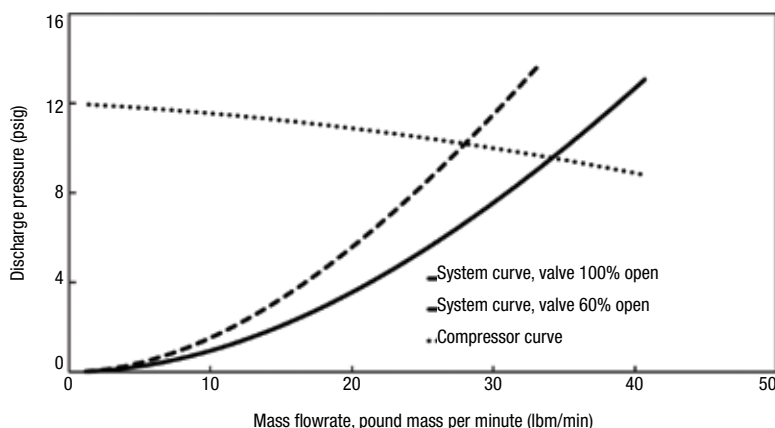
While measuring volumetric flow is a more direct process, there are also measurement tools that yield mass flowrates, such as a thermal mass flowmeter, which determines a mass flowrate based on heat transfer. However, these tools are a less direct way of measuring flowrate and require more careful calibration. Both types of flow specifications provide useful information as long as the implications of each are understood, especially when designing process control schemes.

To remain consistent with the fundamentals, the rest of this discussion will focus on mass flowrate.

Process controls all usually have a similar, high-level pattern. A measuring device reads a certain parameter, and a signal is sent to compare the actual reading with a desired setpoint. The difference between the two causes an actuator to trigger a change in the process. In flow controls specifically, there is communication to a valve actuator that will open and close the valve to adjust the fluid flow.

While this is a common operation, the reason for why this works is not always obvious. If a compressor runs at a constant speed, you might expect the flowrate to also be constant. According to the law of conservation of mass, a constant flowrate will remain no matter what other changes are made to the system. By closing a valve, the area through which the fluid flows is made smaller, and thus, an increase in velocity is observed to keep that same flow.

But it is important to understand that centrifugal compressors can generate different steady-state flowrates with one motor speed. They operate on a curve, similar to a pump. By closing the valve, you have increased the frictional losses in the system, and thus the compressor will need to supply more pressure to overcome those losses.



**FIGURE 3.** Compressors supply energy in the form of flow (kinetic energy) or pressure (potential energy). This concept can be illustrated with a compressor curve versus a system curve with manual valves

This increase in pressure is coupled with a decrease in flowrate. That is why compressor curves are downward-sloping. A compressor supplies energy in the form of flow (kinetic energy) or in the form of pressure (potential energy). Increasing one decreases the other (Figure 2).

It is easiest to show this concept with a compressor versus a system curve for a simple system (Figure 3). The system curve represents the pressure demanded by the system at a range of flowrates. The intersection of the compressor and system curve is the operating point. Increasing the frictional losses in a system will move the system curve leftward (System curve — Valve 60% open) to a new operating point. There is more pressure demanded by the system to ensure that flow occurs because now there are more losses. This example is presented as the control of a manual throttling valve, to help simplify the system behavior and understand the role of component losses and flowrate. (A control valve similarly controls flow based on its pressure-loss information, but the generation of a system curve to visually represent flow control becomes difficult because the valve changes its loss information across the varying flowrates to meet its setpoint.)

Of course, centrifugal compressors are not the only way to move gases. Reciprocating compressors are also common, and they operate at a single flowrate on a near-vertical compressor curve. In the case of reciprocating compressors, the flowrate is not controlled via typical means. Rather, you will only change the operating pressure by the changing losses through the system. If you want to change the flowrate, you will have to modify the compressor directly.

It is also common for steam systems to

not have a compressor at all, where the flow travels from a boiler to a lower temperature and pressure trapping. In this case, while there is no compressor to operate on a curve, the flowrate is still determined by the frictional losses in the system. It can be helpful to define these losses as “resistance,” with an indirect relationship with flow. The more resistance in a system, the less flow will be generated.

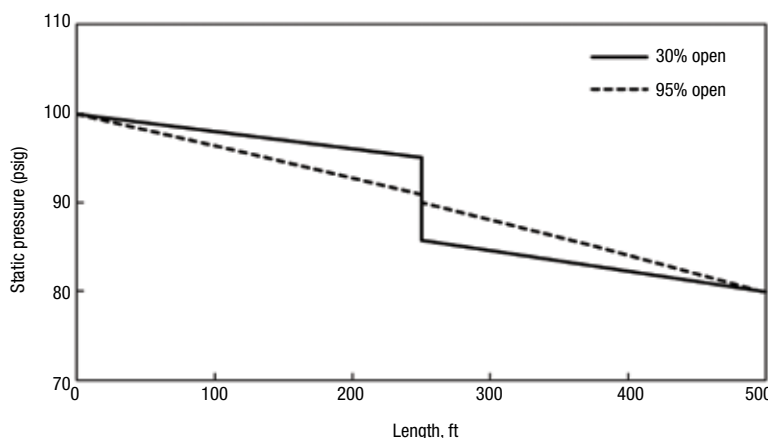
Now that the basics of flow control have been discussed, let’s get into a compressible-flow-specific topic: sonic choking. Most process engineers do not instigate choked flow on purpose, but it is a form of controlled flow, nonetheless. Sonic choking occurs when the velocity of the fluid is the same as the local speed of sound (that is, having a Mach number of 1). This tends to occur at restrictions in the pipe, such as control valves. Aside from flowing through a special converging-diverging nozzle, this is the limit of flow. No matter how low you drop the downstream pressure,

you will not drive any more flow. It is very important for engineers to keep an eye on gas velocities through control valves because they may be approaching that choke point.

### Pressure control

It was previously mentioned that a pressure differential is the driving force for flow. In any case other than choked flow, engineers can control flow by controlling the pressure in the system. Although the goal may be to control flow, keep in mind that an operator actually controls the driving force behind it, not the end-controlled variable directly.

Perhaps an engineer is more interested in controlling pressure than controlling flow. That is, pressure becomes the controlled variable. When dealing with a steam system, you may want your pressure to remain low enough to avoid condensation and slug flow in your lines. Maybe you are interested in keeping a high-enough pressure downstream to avoid high velocities and their accompa-



**FIGURE 4.** The graph shows the pressure profiles for two cases, one in which a valve is more open (dashed line) and one with the valve more closed (solid line). The pressure drop across the valve is higher with the more closed valve, but the starting and ending pressures are the same

nying noise levels.

Valves are a source of pressure loss in the system. This gives engineers a solid foundation to design, and they can use that to their advantage to throttle flow. In most processes, there are two main categories of pressure control valves: pressure-reducing and pressure-sustaining. Relief valves are a common component in gas handling, but they are meant more for safety precautions than for controlling pressure. Pressure-reducing valves control the pressure downstream, while pressure-sustaining valves control pressure upstream.

There is a unique aspect to this type of control. Many control valves operate on a pneumatic or hydraulic pressure signal from the controller for actuation. In this special case, pressure is both the controlled variable and the actuating signal, allowing design for a self-actuated pressure control valve. These do not go through a typical control loop with an outside signal to a controller. In industry, self-actuated pressure control valves and pressure regulators are used synonymously. Not all pressure-control valves are self-actuating, but it is helpful to understand the potential for process simplification.

Pressure-reducing valves exercise control by closing to create more frictional loss and pressure drop, or by opening to do the opposite. A pressure-sustaining valve will operate similarly (by opening and closing), but for controlling the upstream pressure. Many people understand pressure-reducing valves because valves are said to cause pressure drop. But the flipside to that pressure drop is that a higher pressure is sustained upstream. The same opening and closing can achieve both goals.

This is an easier concept to grasp when

looking at an example of flow going from a 100-psig tank to an 80-psig tank, with a valve directly in the middle of a 500-ft line. Figure 4 shows the pressure profile of two cases. When a valve is in a more closed position, there is more pressure drop across the valve; there is less pressure drop with an open valve. However, the flow from tank to tank will always have

a profile going from 100 psig to 80 psig, no matter what losses are present in the line.

By closing a valve, you both decrease the downstream pressure and increase the upstream pressure. It is up to the engineer to decide what is the controlled variable of interest. Don't forget that there will be less flow in the scenario with a more closed valve; there is more resistance in that line. When you see a pressure profile such as this in a gas-handling process, also keep in mind that density, velocity, volumetric flowrate and temperature are also changing down the line, even though mass flowrate is constant.

The other common type of pressure valve is a relief valve. This type of valve does not typically operate as part of a control loop. However, it is not unheard of for relief valves to be actuated by an outside controller communicating with a separate part of the process. Most often, pressure-relief valves are mechanically operated valves that crack open when the upstream pressure is past a set threshold. This valve relieves an upstream pressure, but it is not meant to control the pressure to a certain setpoint.

Relief valves are used more as safety devices in gas handling processes. With the multi-faceted coupling of parameters in compressible flow, pressure can change for many different reasons. This extra complication makes controlling pressure a more difficult task, and thus the common remedy to pop open valves when pressure gets too high.

The traditional relief valve operates on a pressure differential across the valve. If the difference is greater than the setpoint, the valve will crack to relieve upstream pressure. A balanced relief valve is the kind that operates only on the upstream pressure, not a differential. It has means of cushioning the

effects of back-pressure on valve operation. This type of valve can be very useful when a process requires precise pressure relief upstream of the valve.

A loud whistling sound is almost always experienced with a relief valve cracking. Of course, it depends on what pressure magnitudes are being relieved. With non-hazardous process gases, relief valves often vent to atmosphere. That can result in quite a large drop in pressure from the process line. This extremely low pressure produces a large driving force for flow. And that flow moves out of the valve with a very high velocity. When venting to such an open space, there is a major drop in the potential and kinetic energy of the fluid. As a result, the gas cools, slows down and drops pressure.

Since energy cannot just disappear, the question becomes “where does it go?” Some of the energy goes into the volume expansion, and some goes into producing sound. A common safety concern with valves is sound levels. In fact, this is not just the case with relief valves, but with any type of valve. By dropping the pressure and increasing the velocity, you will hear an accompanying sound. Some of the pressure loss gets converted into sound, which is just the propagation of a pressure wave. The level of that sound depends on the velocity, flow area, pressure differential, and the mitigation efforts in place. This aerodynamic noise has been the cause for OSHA safety guidelines (Section III: Chapter 5) to protect the hearing of those in the field.

### Controlling the driving force

There are many factors to keep in mind when designing for flow or pressure control in gas handling processes. Pressure is the driving force of the system, even when you are designing for flow control. By changing the frictional losses in the system, you change the operating mass flowrate. Mass conservation is a fundamental law, but volume conservation is not. Volumetric flow, velocity, density, and temperature will all change according to the fluid’s equation of state even though mass flow may be constant. Down the length of an adiabatic and constant cross-sectional area pipe, you will see pressure and density decrease, volumetric flowrate and velocity increase, and most often will see temperature drop.

When pressure is your controlled variable, you affect both the upstream and downstream pressure (and flow) with the same valve operation. Closing a valve will

create larger pressure losses, which implies a greater upstream pressure, a lower downstream pressure, and a lower mass flowrate. A pressure-reducing valve controls the downstream pressure, while a pressure-sustaining valve controls upstream.

Depending on the process application in question, engineers design a wide range of flow and pressure control methods, but it all boils down to changing the pressure to change the momentum transfer. That is the beautiful consistency of chemical engineering: there is always a driving force behind transport phenomena. Controlling that driving force is how you ultimately control your process variables. ■

*edited by Scott Jenkins*

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# Rare Noble Gases: Production and Use of Ne, Kr and Xe

Rare noble gases neon, krypton and xenon have unique properties and can be obtained from air, but the economics of isolating them at air-separation units depends on demand and pricing. This article provides information on their production and use

**Jim Dray**  
JRD Gastech LLC

## IN BRIEF

BASICS OF AIR  
SEPARATION

ECONOMICS OF  
PRODUCTION

NEON PRODUCTION

KRYPTON AND XENON  
PRODUCTION

The term “rare gases” typically refers to the elements neon (Ne), krypton (Kr) and xenon (Xe). They are noble gases that are collected and separated by cryogenic separation of air, the primary method of producing two of the world’s largest commercial chemicals (oxygen and nitrogen). The concentration in air of Ne, Kr and Xe total less than 20 parts per million (ppm). Argon (Ar) is also a noble gas recovered from air, but its concentration in air is comparatively much higher (0.93%) and is not considered rare. Helium (He) is present at about 5 ppm in air, but is much more easily recovered from certain natural gas deposits, and is not usually commercially recovered from air. All noble gases lighter than radon (He, Ne, Ar, Kr and Xe) are valued for their inertness and other unique properties. This article provides information about the separation of Ne, Kr and Xe from air and their respective uses.

### Basics of air separation

Clean, dry air is made up of approximately 78% nitrogen and 21% oxygen, 0.93% argon and trace amounts of other components. The concentrations of other impurities (water, carbon dioxide, hydrogen, nitrous oxide, methane, ethane and miscellaneous hydrocarbons) are a function of ambient conditions and local circumstances.

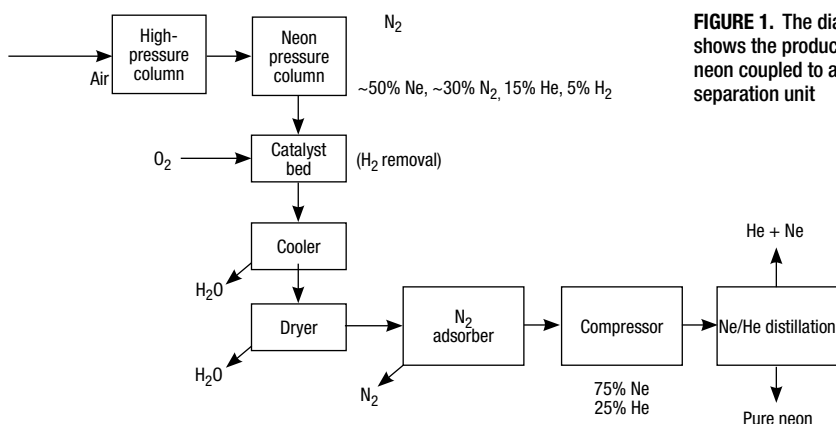
Production of oxygen and nitrogen from air with a cryogenic air separation unit (ASU) is typically done in two distillation columns operated around  $-300^{\circ}\text{F}$  ( $-185^{\circ}\text{C}$ )  $\pm 20$  degrees. The low temperatures are required to convert air, oxygen and nitrogen from gas to liquid. Liquid contacting vapor is what makes the distillation columns work. The two columns

are typically built with a low-pressure column that sits above a high-pressure column. The stacked configuration allows for efficient heat exchange between liquid at the bottom of the low-pressure column and vapor at the top of the high-pressure column.

The distillation columns, heat exchangers and cold piping are all enclosed in a “cold box” that is filled with perlite insulation to minimize heat leaking into the system. The low-pressure column typically operates at around 5 psi at the top of the column. The high-pressure column operates at around 60 psi. The higher pressure is required to raise the boiling point temperature of nitrogen enough that it condenses at a higher temperature than the liquid oxygen, boiling at the bottom of the low-pressure column. This occurs in a heat exchanger (known as the main condenser) located in the bottom of the low-pressure column, where the liquid oxygen collects.

The high-pressure column separates the nitrogen from the oxygen, producing high-purity nitrogen (typically with single-digit-parts-per-million levels of  $\text{O}_2$ ) at the top of the column and a low-purity oxygen stream at the bottom (35 to 40%  $\text{O}_2$ ). Roughly 45% of the air feed to the high-pressure column is removed from the top of the high-pressure column and sent to the top of the low-pressure column as reflux liquid, or as nitrogen product. The rest is returned to the high-pressure column as reflux to achieve the desired ppm- $\text{O}_2$  level in the nitrogen.

Oxygen is produced from the bottom of the low-pressure column. High-purity nitrogen can be produced from the top of either or both columns. For large volumes of nitrogen (more than 10% of the feed air or half the oxygen flow), the top of the low-pressure col-



**FIGURE 1.** The diagram shows the production of neon coupled to an air-separation unit

umn is usually the economic choice. Taking gaseous nitrogen from the top of the high-pressure column would reduce the liquid available for reflux to the low-pressure column, reducing oxygen recovery.

Before the air is fed to the distillation columns, it must be compressed, purified and cooled. This prevents impurities from freezing in the heat exchangers, piping and columns, and thus avoids plugging the equipment, which would lead to poor performance and possibly unsafe conditions.

Once the purified air begins its journey through the cold box, the incoming air is cooled further via heat exchangers that transfer the warm air's heat to the outgoing cold products. The incoming air gets colder and the outgoing products that have been processed are warmed to near ambient temperature. This heat exchange activity minimizes the total energy required, but there are still losses and heat leaks into the system.

The two main methods of providing refrigeration to overcome losses are liquid addition (liquid oxygen or liquid nitrogen from a tank or separate liquefier) and gas expansion. Gas expansion can be done as part of the ASU system, in the same cold box. It is done with a turbine, which spins at high-speed as the gas passes through it and expands. Air and nitrogen are the two fluids that are typically used in turbines.

Argon can be recovered as a valuable byproduct from an ASU that produces oxygen by adding one or more additional columns to the system.

The boiling points of each of the rare gases determines where in the air separation process it is recovered. The normal boiling point of Ne is 27.1K, warmer only than He and H, which are also present in air. These three components

concentrate with the nitrogen streams at the top of the columns, where nitrogen is the coldest stream of the three major components. The three lowest-boiling-point gases are commonly referred to as noncondensables, because they will concentrate if the process is not designed to remove them before they build up to unacceptable levels.

### Economics of production

Ne, Kr and Xe exist in air at low levels (18.2, 1.14 and 0.087 ppm, respectively)

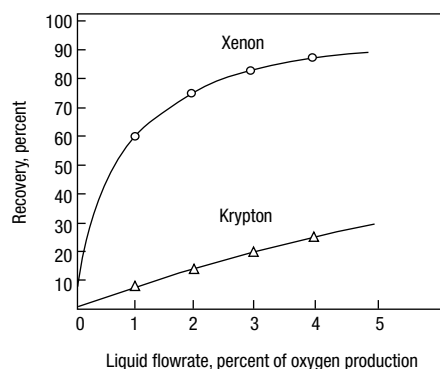
and are largely ignored in most ASU design calculations. The volumes in Table 1 illustrate what is contained in the air feeding an ASU that produces 100 tons per day of oxygen. This is a common size for a plant that could easily make all of its oxygen product as a liquid. If all product was produced as liquid, it would be over 7.6 million gal/yr. At 100% recovery, the Ne production would be less than 400 gal/yr, while Kr would be around 50 gal/yr and Xe just under 5 gal/yr. Rare gases are typically sold as gaseous liters at standard conditions. One liter equals 0.001 of a cubic meter, which tends to make the volumes seem larger.

The potential amount of rare gases that could be produced from a 100 ton/d ASU plant is too small to support the capital needed for systems needed for the isolation of rare gases. However, rare gases can be valuable byproducts, so it can be attractive to collect and refine one or more of them at large ASUs.

Product demand and projected pricing determine at which point it becomes economically attractive to include rare gases as part of the product slate. A air-separation plant that produces nominally 1,000 ton/d of oxygen is usually the point rare gas recovery starts to become attractive. New ASUs are being built as large as 5,500 ton/d (5,000 metric tons per day).

Historically, the prices of rare gases have been quite elastic with supply and demand. When demand increases, the prices increase and more capacity is added to the market, which causes downward pressure on the price. This situation was evident in 2015, when a variety of factors disrupted the Ne market, causing prices to rise by a factor of six or more. Prices have since returned to historical levels.

The decision to produce rare gases often relates to the value of the downstream product, since oxygen (or nitrogen, or both) often represents only a few percent of the raw material costs to make the final product. Producing rare gases may not have enough impact on the cost to of the final product to justify any additional costs or risks associated with including them. The risks include increased downtime, or, in some cases, extended, unplanned shutdowns. At this time, it is not economically viable to design, build and operate a plant that produces one or more rare gases and



**FIGURE 2.** The graph shows the impact of O<sub>2</sub> withdrawal rate on the recovery recovery of Xe and Kr from the reboiler sump

vents the oxygen, nitrogen and argon. There is potential to recover rare gases from other chemical processes that use large volumes of air, like some ammonia processes, but it has not proven to be economically viable yet.

### Neon production

Removing non-condensable gases is important to the design of an ASU. One of the key components of an ASU is the main condenser that operates as the reboiler at the bottom of the low-pressure column and the condenser at the top of the high-pressure column. Minimizing the temperature difference between the top of the high-pressure column and bottom of the low-pressure column has a direct impact on the air pressure required to drive the process, as well as its separation power. In some processes, a non-condensable-gas vent is used to remove the non-condensables before they build up to unacceptable levels. It is desirable to remove as little nitrogen gas along with the non-condensable gases as is practical, because removing nitrogen-rich gas from the top of the high-pressure column decreases the liquid nitrogen reflux sent to the top of the low-pressure column. Oxygen and argon recoveries typically improve as liquid nitrogen reflux is increased. Process cycle selection has a huge impact on Ne recovery potential. In some processes, significant fractions of the feed air may bypass the high-pressure column, reducing the Ne available for recovery. Some processes produce refrigeration by expanding a portion (nominally 10%) of the vapor air feed directly to the low-pressure column.

Many modern processes send a significant fraction of the air feed as liquid

directly to the low-pressure column. These features can reduce neon recovery by 20% or more. Other processes remove nitrogen gas from the high-pressure column, removing Ne, He and H<sub>2</sub> at the same time. When significant quantities of nitrogen gas are removed from the top of the high-pressure column, the liquid nitrogen reflux to the low-pressure column is removed a few stages below the top of the high-pressure column, significantly changing the concentration of non-condensables at the top of the high-pressure column. If the reboiler is designed for non-condensable recovery, the Ne will concentrate to roughly 1% in the non-condensable vapor stream, along with most of the He and H<sub>2</sub>. The remainder of the stream is nitrogen.

When the low-pressure column reboiler design makes concentrating the non-condensables to high levels impractical, a stripping column can be used to collect Ne. The liquid nitrogen that flows from the high-pressure column to the low-pressure column as reflux is passed through a stripping column on the way to the low-pressure column. The Ne-rich vapor stream is withdrawn from the top of the column. The nitrogen liquid headed to the low-pressure column as reflux is withdrawn from the bottom of the stripping column (Figure 1). The stripping column can improve reboiler performance because the non-condensables do not concentrate to such high levels, minimizing the apparent temperature difference across the reboiler, and increasing Ne recovery. This reduces pressure in the high-pressure column and air compressor power.

In either case, the Ne-containing stream is at the pressure close to that of the high-pressure column (nominally 60 psi). It is fed to a column/condenser system, where it is condensed against low-pressure (1 psi) liquid nitrogen at 78K. This will concentrate the Ne up to around 50%. A potential improvement to this operation is to use a vacuum pump to reduce the liquid N<sub>2</sub> boiling temperature to just above nitrogen's freezing point (63.2K). Reducing the condensing temperature reduces the N<sub>2</sub> content in the crude Ne stream.

Crude neon is typically shipped by tube trailer to a central processing facility, so transportation, equipment and operating cost tradeoffs are used to evaluate whether or not a vacuum pump system is desirable.

A catalyst bed operating above ambient temperature is typically used to react hydrogen from the stream, with oxygen added as required to feed the reaction. Adsorption is typically used to remove the water and most of the remaining nitrogen and oxygen, yielding a crude neon stream that contains roughly 75% Ne and 25% He. The compressed, crude Ne stream is then cooled to around the freezing point of N<sub>2</sub>, where it is passed through a trap (adsorber bed) to remove trace concentrations of oxygen and nitrogen that would freeze when the Ne-rich stream was cooled close to its dew point. The cooled Ne/He mixture is fed to a small distillation column, where the pure Ne collects in the bottom as liquid and the He, trace H<sub>2</sub> and some Ne leave from the top of the column. The Ne product can be recovered as liquid or gas from the column.

If it is recovered as liquid, then the refrigeration source for the top condenser has to be sized to provide the refrigeration needed. Mechanical refrigeration and liquid H<sub>2</sub> are two potential refrigeration sources. The column pressure is an important variable for designing the separation. Increasing column pressure can minimize the Ne lost out the vent with the He. It also increases the temperature difference between the boiling and freezing points of Ne in the process. At atmospheric pressure, the difference between the boiling and freezing points of Ne is only 2.6K. Increasing the column pressure increases the boiling point of Ne and makes the column easier to operate and restart after a shutdown.

### Krypton and xenon production

The boiling points of Kr and Xe are above that of liquid oxygen, so Kr and Xe will concentrate with the oxygen product at the bottom of the low-pressure column. The process and main condenser design have a huge impact on potential Kr and Xe



recovery. Many older designs used oxygen compressors that were fed with gas from the low-pressure column. Reboilers of that era were set in a pool of liquid oxygen that was recirculated and maintained at a high level to ensure that liquid and gas both flow out the top of the reboiler. This minimizes the chance of boiling to dryness and concentrating hydrocarbons or other impurities (CO<sub>2</sub> and N<sub>2</sub>O, for example) to unacceptable levels in oxygen. The primary hydrocarbons in the main condenser of an ASU are methane and ethane, but propane and other trace contaminants are also present. These hydrocarbons have boiling points above oxygen, close to those of Kr and Xe, and present major safety concerns when recovering Kr and Xe.

The older ASU designs have the potential for Kr and Xe recoveries greater than 90% because almost all the liquid flowing down the low-pressure column is vaporized and returned to the column or removed as oxygen product. Kr and Xe could concentrate to roughly 60 ppm each, with zero liquid oxygen withdrawal.

Newer ASU processes, commonly known as liquid pumped cycles, typically remove liquid oxygen (instead of gas) from the low-pressure column. The liquid oxygen withdrawn from the low-pressure column is typically pumped to the desired pressure and vaporized against high-pressure air in the main heat exchanger. This approach eliminates the oxygen compressor in most applications, but makes Kr and Xe recovery more difficult, because the Kr and Xe in the sump liquid is roughly 5.5 ppm Kr and 0.4 ppm Xe.

In new designs without Kr and Xe recovery, the liquid oxygen is withdrawn from the sump of the high-pressure column, where the main condenser is located. This results in lower concentrations of hydrocarbons and other impurities in the sump liquid, but also removes all the Kr and Xe from the process. It is

possible to recover most of the Kr and Xe with other methods, but they increase process complexity and costs.

One approach to achieve nominally 70% Xe recovery and lower Kr recovery is to take the liquid oxygen product from the low-pressure column before it reaches the main condenser. The liquid-to-vapor ratio at the bottom of the column varies with the chosen cycle, but is close to 1.4 to 1 in most cases. The liquid oxygen removed as product never reaches the reboiler, so the portion of the Kr and Xe in the air feed is lost. It is much easier to recover Xe than Kr with this type of process. Figure 2 (from U.S. patent 5,069,698) illustrates the impact of the liquid O<sub>2</sub>-withdrawal rate on the recovery of Kr and Xe from the reboiler sump. High Xe recovery can be achieved with a relatively small liquid O<sub>2</sub> feed to a Kr/Xe concentrating column, requiring smaller equipment and less energy than a high-Kr recovery system.

There are other, more complex processes that can be used to recover Kr and Xe from liquid pumped cycles. They tend to have more significant process impact on, and increase the cost of, the ASU process to which they are attached.

Concentration of Kr and Xe is usually accomplished in a series of unit operations. The large difference in boiling points among O<sub>2</sub>, Kr and Xe makes it easy to separate the molecules by distillation. The first step is a short column that raises Kr and Xe concentrations to between 0.1 and 0.5%. The main complication is that the boiling points of hydrocarbons common in the air are close to those of Kr and Xe. The hydrocarbons must be removed or diluted relative to the oxygen before the rare gases can be concentrated further. Another key operation is to remove trace quantities of CO<sub>2</sub> and N<sub>2</sub>O from the oxygen stream. This is most often done by adsorption. CO<sub>2</sub> and N<sub>2</sub>O will reach unacceptable levels in the concentration process if they are not re-

**TABLE 1. UNIT COMPARISON OF CONTAINED FLOWS OF OXYGEN, ARGON AND RARE GASES IN A 100-TONS-PER-DAY OXYGEN PLANT**

Element	Tons per day	ft <sup>3</sup> /hr (at normal temp., pressure)	Liquid gallons per year	Gas liters per year
Oxygen	100	100,600	$7.66 \times 10^6$	$2.50 \times 10^{10}$
Argon	5.5	4,464	$3.48 \times 10^5$	$1.10 \times 10^9$
Neon	0.0055	8.74	396	2,159,048
Krypton	0.0014	0.55	52	135,237
Xenon	0.0002	0.042	4.9	10,321

**TABLE 2. BOILING- AND FREEZING-POINT DATA FOR GASES IN AIR**

Element	Normal boiling point, K	Freezing point, K
Helium	4.2	n/a
Hydrogen	20.3	14.0
Neon	27.1	24.6
Nitrogen	77.4	63.2
Oxygen	90.2	54.4
Argon	87.3	83.8
Krypton	119.8	116
Xenon	165	161.4

moved. These components can solidify in the liquid oxygen and block tubes, heat exchanger passages and/or piping. This creates other serious safety concerns, especially dead-ended boiling in an oxygen-rich environment. Dead-ended boiling has been identified as a cause of major oxygen-plant energy releases (incidents) and must be avoided.

One commercial approach to remove hydrocarbons is to vaporize and warm the oxygen-rich stream to roughly 900°F and send it through a catalyst bed to react the hydrocarbons, consuming a small amount of the oxygen in the stream. However, water and CO<sub>2</sub> created by combustion of the hydrocarbons must be removed before final separations into pure product streams.

An alternative to the high-temperature catalyst method is to replace the oxygen with an inert gas (typically Ar or N<sub>2</sub>), which eliminates concentration of hydrocarbons as a safety concern. At least one additional distillation column will be required without the catalyst system. Adsorption is also used as a purification method for Kr and Xe, but it is most often used when recovering only one of the two heavier rare gases. For the highest-purity products, additional processing may be recovered to remove trace compounds that concentrate with the Kr and Xe, such as CF<sub>4</sub> and SF<sub>6</sub>. They are present in the air at roughly 44 and 3 parts per trillion, respectively, but can concentrate to unacceptable levels when concentrating air components in the ppm or ppb range.

## Commercial uses

Anesthesia using Xe/O<sub>2</sub> mixtures is very intriguing and effective. It has been called a nearly ideal anesthetic gas. Unfortunately, the complexity of recovering what the patient exhales and reprocessing the Xe-rich gas makes it difficult to gain widespread acceptance. Plasma televisions and lasers are two areas where Xe is widely used. In these applications, Xe is mixed with other gases, often Ne and He. Xe thrusters are a valuable technology for deep space flight and other space applications. Ion thrusters issuing pure Xe require much less mass than conventional propellants when using Xe and can achieve higher velocities. With the cost of sending items to space still in the range of thousands of dollars per pound, the high price of Xe is not a significant obstacle.

Other areas where pure xenon is used include high-efficiency lighting in automobiles, computer chip manufacturing, dark matter experiments in physics research and nuclear fuel processing.

Krypton has a relatively high mass for a gas and is an excellent insulator. It has had many uses over the years, filling niche markets, including lighting and window manufacturing. When the cost is low enough compared to Xe, it can replace Xe in some applications. It can also be used as a “green” refrigerant.

Neon is often the main component of laser gas mixtures, and is critically important to the manufacture of high-capacity computer chips. It has a distinctive red glow when energized and is frequently used to make decorative signs. It is an effective refrigerant for applications around 30K. The latent heat per gallon of liquid Ne is more than 40 times the latent heat per gallon of liquid He, making the design of refrigeration systems smaller and simpler. ■

*Edited by Scott Jenkins*

## Author

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# Modular Process Design: Chemical and Thermal Recycling of Acids

In order to recycle spent acids from different applications, a design approach based on different process modules gives operators the flexibility needed to handle different contaminants

**Kevin Schnabel,  
Sebastian Bialek,  
Peter Pataky and  
Max Heinritz-  
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## IN BRIEF

COMPOSITION OF SPENT  
ACIDS

PROCESS MODULES

DESIGN CRITERIA

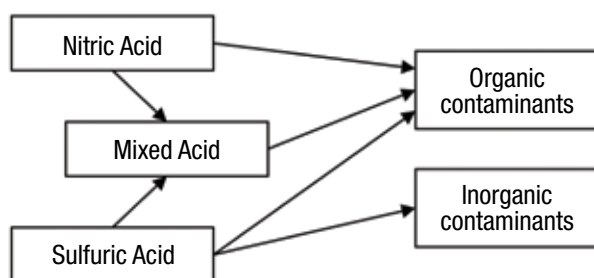
EXAMPLE CASE:  
RECYCLING OF  
NITRATION ACID

FINAL REMARKS

Spent or waste acids (SA) containing sulfuric or nitric acid are a byproduct of various processes involving nitration or sulfonation reactions or of processes, in which these acids are used for leaching or as a catalyst. These reactions and processes are used in the industrial production of polymers, drugs, pigments, other specialty chemicals, explosives, battery chemicals or metallurgical products. The SA from such industries is diluted by reaction water and contaminated with organic and inorganic compounds.

Stricter environmental regulations and rising raw-material prices in recent years have led to considerable cost and organizational effort for the procurement of fresh acid and the disposal of SA. An effective acid-recycling system can contribute to a more cost-efficient, independent and environmentally friendly production process. A challenge for recycling systems, however, is the wide variety of production processes and the resulting SA with partly unknown contaminants.

Chemical and thermal recycling of SA consists of various steps and the respective unit operations. The applicability of these unit operations has to be determined individually, considering factors such as volatility, solubility and reactivity of the known and unknown contaminants, the required quality and concentration of the recycled acid and



**FIGURE 1.** The classification of spent acids can be based on the acid itself and the type of contaminants

the available materials of construction for the treatment of the highly corrosive acids. For an efficient process design, pre-defined process modules can be combined, comprising unit operations for specific cases. With such a modular approach, the thermal SA recycling is divided into three steps: (1) removal of contaminants; (2) separation of acid mixtures; and (3) acid concentration increase and absorption. The used modules



**FIGURE 2.** These vials show the spent acid from a sulfonation process before (left) and after (right) thermal oxidation. The middle vial shows a sample taken before completing the oxidation

include oxidation reactors, precipitation, filtration, distillation and rectification, as well as absorption and stripping equipment.

### Composition of spent acids

Spent acids result from a variety of processes in which nitric or sulfuric acids are used as reactants, leaching solutions or as a catalyst. Depending on the production process, the composition of the SA varies considerably in terms of acid concentration and impurities. Figure 1 illustrates the classification of SA resulting from nitration or sulfonation reactions (Figure 2) and acid leaching based on the containing acid (nitric or sulfuric acid or both) and the type of contaminants (organic or inorganic). While hydrochloric acid is also used in various industrial processes, chemical or thermal recycling of spent hydrochloric acid is applied less often as compared to spent nitric and sulfuric acids and therefore will not be discussed further here.

In industrial production processes involving nitration and sulfonation reactions (for example, in the production of polymers, drugs, explosives or pigments), the SA is contaminated with organic compounds. These contaminants include at least traces of the reactants and product (for instance, nitrocellulose, nitrobenzenes, benzenesulfonic acid, glycol-nitrates, picric acid, nitrotoluenes) but also byproducts. For recycling of these SA, in some cases, a complete removal of certain contaminants is required for safety reasons (such as for explosive compounds) while some contaminants can be recycled to the originating process. Depending on the acid used for the nitration, the SA is either a nitric acid or a mixture of nitric and sulfuric acid with organic impurities.

Spent acids from inorganic industries like the production of inorganic pigments or battery chemicals (for example, titanium dioxide via the sulfate route or lithium refining) or metallurgical industries (such as copper electrolysis, or demister acid from smelter sulfuric acid plants) contain inorganic impurities that could precipitate in the production process and/or affect the product quality.

These inorganic impurities must be depleted or removed entirely.

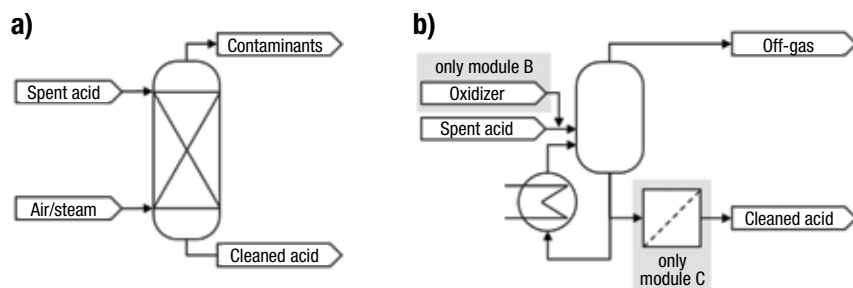
The concentration levels of the acid in the SA range from diluted solutions (below 10 wt.% acid) to high concentrations with partly anhydrous acids.

### Process modules

The treatment of SA applying process modules consists of unit oper-

ations for mechanical separation (filtration), thermophysical separation (evaporation, distillation/rectification) as well as chemical (oxidation) and physicochemical operations (precipitation, stripping and absorption). Together with the auxiliary machines (pumps, vacuum pumps and compressors) and static equipment (heat exchangers, evaporators, tray columns, packed-bed columns, re-





**FIGURE 3.** Shown here are flow diagrams a) for module A (stripping) and b) for the modules B and C (thermal oxidation and precipitation/filtration). The oxidizer is only added in module B and the filtration only used in module C

action vessels) made of corrosion- and temperature-resistant materials these unit operations form process modules that can be combined to treat various SA. In general, each process module serves one of the three above-described steps in the SA recycling process as is further described below. Table 1 shows a summary of the process modules.

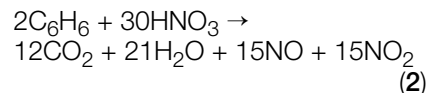
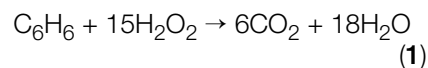
#### Step 1: Removal of contaminants.

The contaminants or impurities found in the SA can be classified as volatile or non-volatile. Modules for the removal of these contaminants are shown in Figure 3. Volatile compounds can be removed from a high-boiling SA by stripping with air or steam (module A). In general, the vapor pressure of many organic compounds (for example, nitrobenzene) and some inorganic compounds (such as hydrogen fluoride or oxides of nitrogen) increases with temperature. Therefore steam stripping promotes the separation. For an optimal gas-liquid contact, stripping is performed in packed bed columns. These columns are made of either glass-/polymer-lined steel,

fiber-reinforced polymer or stainless steel. By stripping these impurities from the SA, it is possible to recover them in a condensation or absorption step downstream of the stripping unit. However, in some cases, recovery is not possible due to a severe safety hazard being posed by some organic nitrates.

Non-volatile or hazardous volatile organic compounds can be destroyed by thermal oxidation (module B). The oxidation must destroy all organics non-selectively to achieve a purified acid and protect downstream equipment from explosive compounds. In order to achieve a complete oxidation, the SA must be heated up to temperatures between 120 and 200°C. For diluted acids whose boiling point is lower than the required decomposition temperature, sulfuric acid can be added to achieve an oxidation in the liquid phase, resulting in smaller equipment sizes. Nitric or mixed acids already contain nitric acid as oxidizing agent, while for SA without nitric acid, an oxidizing agent consisting of hydrogen peroxide or nitric acid, for ex-

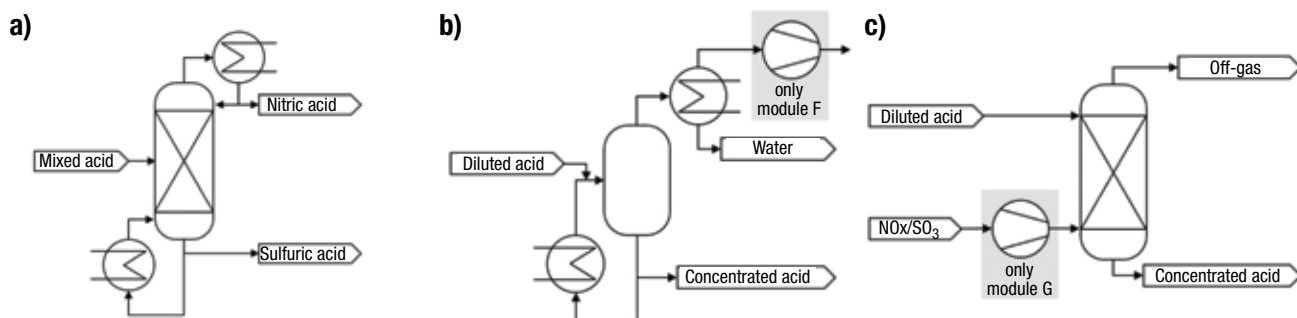
ample, must be added. An example of a reaction of an organic impurity (benzene) with hydrogen peroxide or nitric acid is shown in Equations (1) and (2). Figure 2 depicts a sulfonation SA before and after the thermal oxidation step.



Precipitation and subsequent filtration removes non-volatile inorganic compounds (module C). The precipitation / crystallization is facilitated by increasing the concentration of the inorganic impurities beyond the solubility, which depends on the temperature and acid concentration. This can be achieved by evaporation of excess water and/or cooling of a hot SA to decrease the solubility. A higher acid concentration usually also decreases the solubility of inorganic compounds and therefore has a positive influence on remaining impurities in the system.

#### Step 2: Separation of acid mixtures.

The separation of acid mixtures is done by rectification, as shown in module D (Figure 4a). The higher boiling sulfuric acid is discharged at the column bottom and the nitric acid as distillate from the column overhead. Due to the hygroscopic nature of sulfuric acid, the water from the feed SA is discharged with the sulfuric acid whereby high concentrations of nitric acid can be achieved. The highly



**FIGURE 4.** Shown here are the flow diagrams for various modules. Figure 4a is module D (rectification), in which the nitric acid is separated from the acid mixture as distillate and the sulfuric acid and water as bottoms. Figure 4b is modules E and F (atmospheric and vacuum evaporation) and Figure 4c is for modules G and H (pressure and pressureless absorption). Vacuum pumps and compressors are only used for modules F and G

**TABLE 1. SUMMARY OF PROCESS MODULES FOR THE TREATMENT OF SPENT ACIDS**

Module		Purpose	Mechanism
A)	Stripping	Removal of volatile inorganic/organic compounds	Differences in volatility/vapor pressures of impurities and spent acid
B)	Thermal oxidation	Decomposition of non-volatile or hazardous organic compounds	Oxidation of organic compounds with a oxidation agent at high temperatures
C)	Precipitation / filtration	Removal of non-volatile inorganic compounds	Reducing the solubility of inorganic impurities by lower temperature or increasing acid concentration
D)	Rectification	Separation of acid mixtures	Evaporation of lower boiling nitric acid while diluting the hygroscopic sulfuric acid
E)	Atmospheric evaporation	Concentration increase by evaporation of excess water	Removal of excess water by evaporation
F)	Vacuum Evaporation		
G)	Pressure Absorption	Production of acid from gaseous precursors	Absorption of gaseous acid precursors (NO <sub>x</sub> , SO <sub>3</sub> )
H)	Pressureless Absorption		

corrosive nature of the boiling sulfuric acid-nitric acid-mixture requires glass or glass-lined packed bed columns for the rectification.

**Step 3: Acid concentration increase and absorption.** An in-

crease of the acid concentration is achieved by evaporation (Figure 4b). Sulfuric acid concentrations near the azeotropic point at approximately 98.5 wt.% are achievable by evaporation of excess water. Depending

on the concentration of the feed SA and the targeted concentration of the product acid, evaporators operated under atmospheric pressure (module E) or vacuum (module F) can be used. By combining evaporators at two different pressure levels, it is possible to reuse the evaporated water by vapor compression for the heating of vacuum falling-film evaporators. In vacuum evaporators with an absolute pressure as low as 40 mbar the sulfuric acid concentrations close to the azeotropic point can be achieved at significantly lower temperatures (approximately 225°C) compared to the atmospheric boiling point (approximately 335°C).

Precursors of nitric and sulfuric acid (NO<sub>x</sub> and SO<sub>3</sub>) that are formed during oxidation (as described in Step 1), rectification and evaporation can be absorbed to produce moderate or high concentrated nitric or sulfuric acid (Figure 4c). Absorption of NO<sub>x</sub> produces nitric acid with concentrations close to

**TABLE 2. COMPOSITION OF THE SPENT ACID FOR THE EXAMPLE**

Component	Content
H <sub>2</sub> SO <sub>4</sub>	65 wt. %
HNO <sub>3</sub>	10 wt. %
H <sub>2</sub> O	22 wt. %
Organics	3 wt. %

70 wt.%. Absorption of NO<sub>x</sub> to produce nitric acid involves a gas-phase oxidation of nitrogen monoxide and absorption of nitrogen dioxide in water [see Equations (3) and (4)]. The gas-phase oxidation as well as the dissolution of nitrogen dioxide in aqueous solution is favored by increased gas pressure. The absorption is performed in stainless-steel tray columns.



Evaporation of sulfuric acid above 90 wt.% produces a SO<sub>3</sub>-rich gas. The absorption of this SO<sub>3</sub> increases the concentration of sulfuric acid. The absorption does not necessarily require an increased gas pressure and can be performed in the same pressure system as the initial evaporation step.

### Design criteria

The selection of the process modules is determined by the composition of the SA and the required concentration and quality of the product acids. For the removal of contaminants, modules A to C are selected depending on the type of contaminant. To determine the adequate design parameters for these process modules, depending on the SA to be treated, laboratory tests or even pilot plant tests may be required prior to a commercial design. If a separation of a SA consisting of nitric and sulfuric acid is necessary, the process module D is used. Depending on the target concentration of the sulfuric acid product, the evaporation modules E or F are applied or combined. With the aim of achieving high energy efficiency within the system, vacuum evaporation (module F) is used when very low (<15%) concentrated acid must be recycled or high sulfuric acid concentrations shall be achieved. The absorption modules are used for internal recovery of NO<sub>x</sub> (module G) or SO<sub>3</sub> (module H) whenever large quantities of these gases are produced. An

efficient energy recovery is achieved by using the energy provided in the previous process step (for example, thermal oxidation) in the subsequent steps (for example, evaporation). The sequential design of process steps also protects sensitive downstream equipment by initially removing contaminants.

### Example: Recycling of nitration acid

As an example, a recycling process for the treatment of nitration SA is shown in the following. It combines various process modules to produce concentrated nitric acid of 98.5 wt.% and sulfuric acid of 96 wt.% from a SA. The SA originates from nitroglycerine production with the composition shown in Table 2. The process design shown in Figure 5 considers a mass flowrate of 1,000 kg/h SA. All product acids are exported with 40°C.

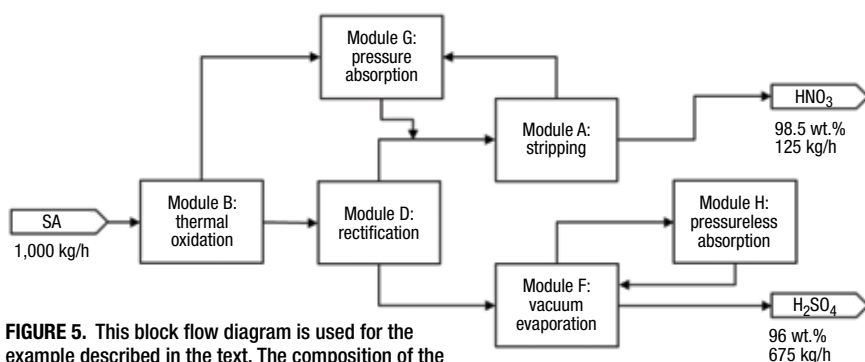
The illustrated exemplary process comprises the decomposition of organic contaminants by thermal oxidation and a subsequent rectification to separate the acid mixture. The nitric acid is separated as distillate and cleaned from volatile NO<sub>x</sub> by stripping to produce a clear acid. The NO<sub>x</sub> are recovered as nitric acid in a pressure absorption. The sulfuric acid from the rectification column bottom is concentrated to 96% by vacuum evaporation while emerging SO<sub>3</sub> is recovered as sulfuric acid.

Heating, evaporation, condensation and cooling in the thermal recycling process requires heating and cooling energy. This exemplary process requires approximately 460 kW heat and 480 kW cooling energy to produce 125 kg/h of concentrated nitric acid and 675 kg/h of sulfuric acid. Besides the positive environmental effect of recycling, the feasibility of recycling SA rather than disposing it and buying fresh nitric and sulfuric acids from the market has to be evaluated by plant operators who are considering a financial investment into a SA recycling plant. Due to the high costs for SA disposal and fresh acid procurement, recycling can significantly reduce the operating expenses (opex) for any production process where great quantities of SA arise.

In order to determine the financial viability of such a capital investment project, the plant operators will have to perform a comprehensive financial assessment study that considers the initial capital expenses (capex) and location-specific opex including energy cost, disposal cost for hazardous waste, product acid pricing.

### Final remarks

In order to recycle spent acids from various applications, a flexible design approach, applying process modules, enables operators to deal with spent acids containing different contaminants. The overall process design is combining independent process modules to an overall recycling process that can recycle different SA with varying acid concentrations and containing organic or inorganic contaminants to achieve different acid concentrations and purities depend-



**FIGURE 5.** This block flow diagram is used for the example described in the text. The composition of the spent acid (SA) is shown in Table 2

ing on the specific requirements. An efficient energy recovery within the process can be achieved, thus enabling significantly reduced opex as compared to the disposal of SA as a hazardous waste and buying of fresh acid from the market. With increasing prices for fresh acids and more stringent environmental regulations applying to the disposal of SA, chemical and thermal recycling of SA becomes increasingly beneficial for plant operators. ■

*Edited by Gerald Ondrey*

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Note: For more on acid recovery, see part 2, "Acid Recovery and Recycle Technologies," on pp. 46–51, as well as the Newsfront, "Acid Recovery Becomes the Norm," *Chem. Eng.*, October 2018, pp. 14–19).



# Acid Recovery and Recycle Technologies

This overview of a number of different methods for handling spent acids will help operators select the technology most suited for a given application

**Bryan and Jared  
Cullivan**  
Beta Control Systems

## IN BRIEF

SULFURIC ACID  
RECOVERY:  
CRYSTALLIZATION

HYDROCHLORIC  
ACID RECYCLE —  
PYROHYDROLYSIS

HYDROCHLORIC ACID  
RECOVERY

MIXED ACID (HF/HNO<sub>3</sub>)  
RECOVERY

The most common pickling, leaching and passivation acids are the mineral acids: sulfuric, hydrochloric and nitric. Mixtures of hydrofluoric acid and nitric acid are also used in cleaning, etching, passivating and chemical milling. Although the organic acids like acetic, phosphoric, citric, oxalic and tartaric have been used in small quantities and in limited applications, the mineral acids continue to dominate. In production uses of highly corrosive mineral acid, the rate of reaction slows as metal oxides dissolve into soluble salt. When the acids wane and reach the end of their effective life, they are either: 1) discarded as a hazardous waste; 2) neutralized into a solid waste sludge; or 3) processed through a recovery system to reclaim the valuable remaining acid. Focusing on the latter, this article describes the operation of the most prevalent acid-recovery and recycle technologies in use today. These include the following:

- Sulfuric acid recovery — low temperature crystallization (Figure 1)
- Hydrochloric acid recycle — pyrohydrolysis (spray roaster/fluidized bed)
- Hydrochloric acid recovery — vacuum evaporation, acid sorption, diffusion dialysis, chemical replacement
- Mixed acid recovery (HF/HNO<sub>3</sub>) — acid sorption, vacuum evaporation, pyrohydrolysis, nanofiltration



**FIGURE 1.** Crystallization is one way to recycle spent sulfuric acid and recover iron

## H<sub>2</sub>SO<sub>4</sub> recovery: Crystallization

Pickling and etching of steel and copper is usually performed in a 12–24% acid solution at bath temperatures between 55°C (130°F) and 75°C (167°F). Sulfuric solutions for electrocleaning and ore leaching are usually kept close to 40°C. The higher solubility of many metal salts in a heated solution allows a saturated solution to be chilled, creating hydrated crystals in the chilled solution. The crystals are commonly separated and collected from the chilled solution through centrifugation or gravity filtration.

Figure 2 shows a solubility curve for iron in various concentrations of sulfuric acid as a function of temperature. Such curves are needed for the design of a crystallizer plant. The process flow diagram shown in Figure 3 depicts a typical sulfuric acid crystallizer used in a steel pickling plant. For the example shown, the spent acid contains a high concentration of iron. Precipitating about half of this iron as ferrous sulfate heptahydrate (FeSO<sub>4</sub>·7H<sub>2</sub>O) produces a recovered acid that can be reused in the pickling plant.

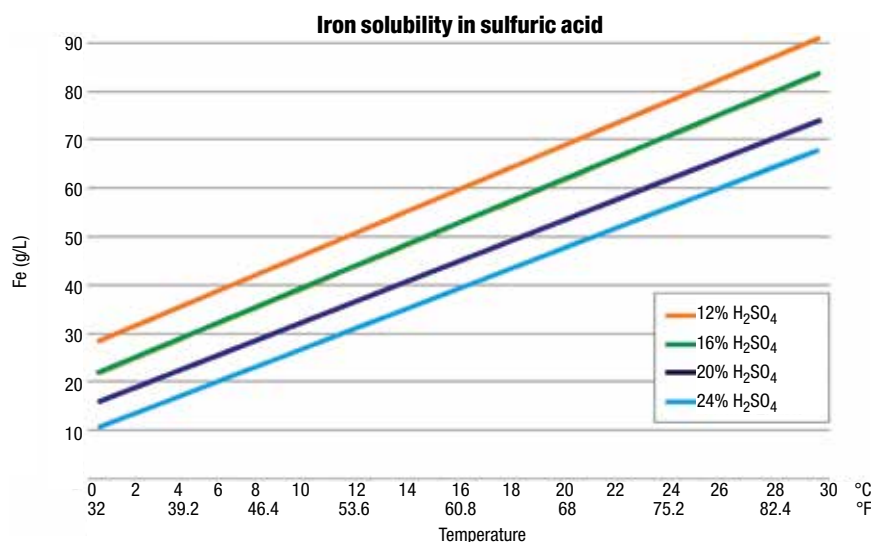


FIGURE 2. The solubility of iron in sulfuric acid varies with concentration and temperature

The total power consumed in this operation of the recovery system approaches 41 kW/ton of spent acid processed. The use of a plate-and-frame (liquid/liquid) heat exchanger reduces the crystallizer energy demand by more than 30%.

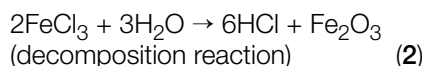
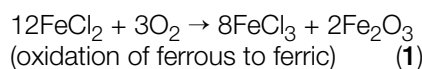
Both batch and continuous crystallization processes use fluid transport, filtration, mixing, heat exchange, refrigeration and centrifuge (screen) separation to achieve the recycle of sulfuric acid.

Ferrous sulfate heptahydrate and copper sulfate tetrahydrate crystals recovered in the process are sold as co-products. Thousands of tons are produced each year and used in agriculture, wastewater treatment and mining. The low operating cost of an acid recovery crystallizer and the value of the co-product make this technology preferable to disposal and neutralization.

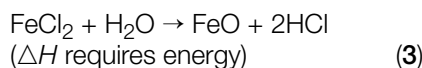
### HCl recycle: Pyrohydrolysis

Differing from the other technologies covered in this article, pyrohydrolysis is a recycle (or regeneration) rather than a recovery technology. Originating from the thermolytic technology used in mining, pyrohydrolysis has been used since the 1950s to recycle spent hydrochloric acid from large steel operations. Both fluidized bed and spray roasting thermally break the metal chloride bond and create metal oxide and HCl products. The metal oxides are collected

as beads or powder at the bottom of the roaster. The resulting HCl gas is mixed with water to form a near azeotrope of HCl after absorption and rectification.



A simplification of the half reactions is given by Equation (3):



In a pyrohydrolysis process, such as that shown in Figure 4, spent acid travels through an evaporative step

before entering the roaster. This pre-concentrated iron chloride solution is then pressure-injected tangentially into a ceramic-lined roaster operating at 550–700°C. The first step of the reaction is the oxidation of ferrous chloride to ferric chloride in the presence of oxygen in the combustion gas. The ferrous chloride concentrate oxidizes and decomposes to a ferric oxide ( $\text{Fe}_2\text{O}_3$ ) solid that is constantly removed from the bottom of the roaster. The HCl gas and water generated in the reaction exit the roaster to be scrubbed and concentrated to a near azeotropic HCl concentration in the absorber/rectifier column. As reported by a manufacturer, pyrohydrolysis requires 50 therms of natural gas to process one ton of spent acid. The process recycles 90+% of the original fresh acid back into good-quality pickling acid.

The ferric oxide co-product of some fluidized bed systems can be re-melted into steel. If the contaminant levels are low, the  $\text{Fe}_2\text{O}_3$  powder from the spray-roasting method can also be used in pigment, ferrite magnets, cosmetics and polishing applications. Environmental monitoring of the process includes accidental emissions and release of iron oxide dust, HCl vapor and chlorine.

### HCl recovery

When a large portion of the hydrochloric acid has been consumed in a pickling or leaching process, the bath is considered spent. If the me-

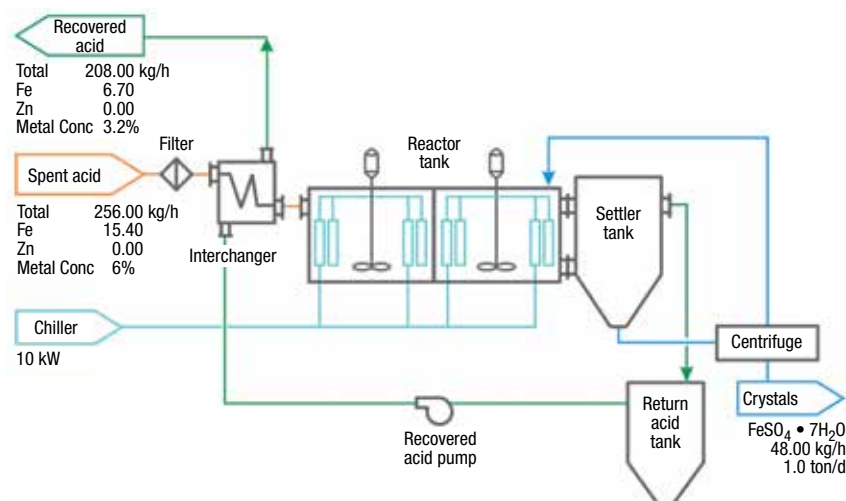
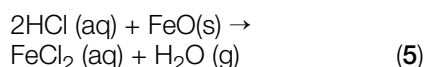
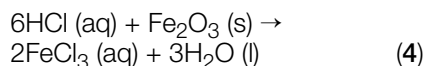


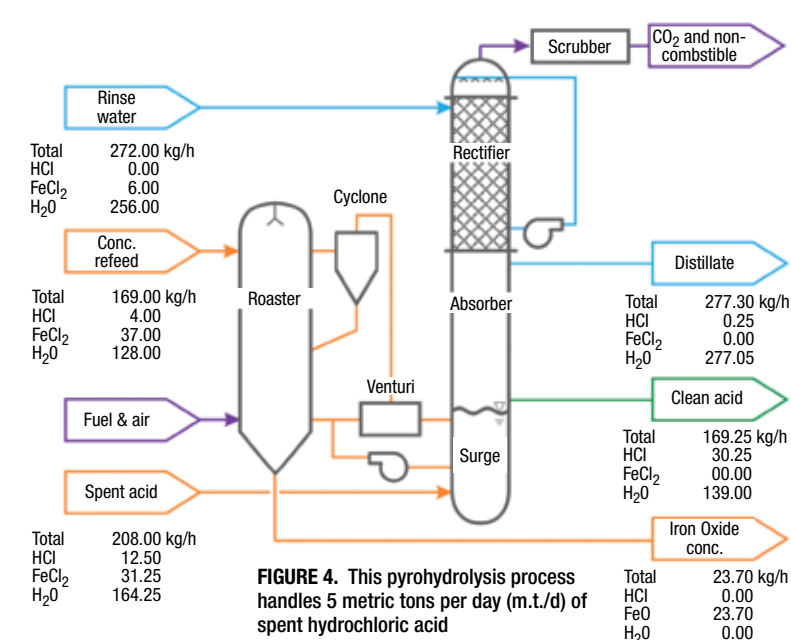
FIGURE 3. Spent sulfuric acid from pickling operations can be recycled using a crystallization process

dian starting HCl concentration for the bath is 15%, the median concentration of spent acid would average 7%. The Fe concentration in the spent acid varies based on the type of rust dissolved. The reactions below depict oxidized rust (orange) and an FeO (green) surface rust in steel processing.



Since most of the iron in spent acid solutions tests as Fe (II), the second equation predominates.

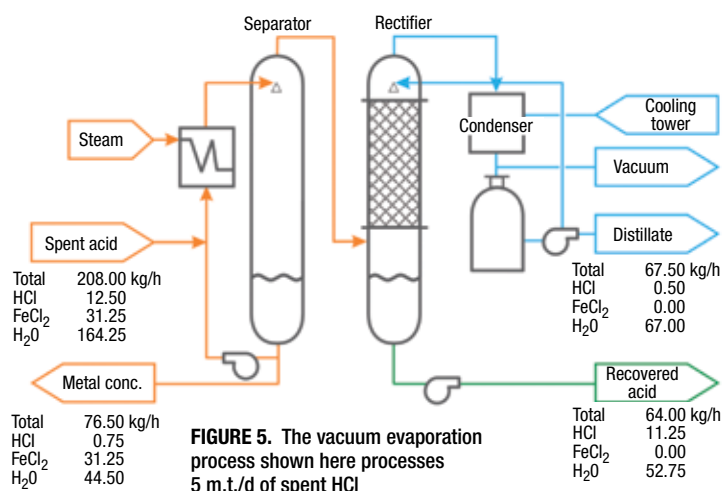
Some production plants are designed with several large pickling tanks to allow extended pickling time. The primary purpose of this is to consume as much HCl as possible before disposal or treatment. Another method to maximize acid



**FIGURE 4.** This pyrohydrolysis process handles 5 metric tons per day (m.t./d) of spent hydrochloric acid

usage is employed by many push/pull sheet and coil pickling operations. The continuous strip steel travels countercurrent to the flow of hydrochloric acid, cleaning the

surface of the steel. The final countercurrent pickle tank is heated to increase the reactivity and consumption of the acid. In contrast, most wire, tube and metal finishing



**FIGURE 5.** The vacuum evaporation process shown here processes 5 m.t./d of spent HCl

plants choose to dispose, treat or recover their acid rather than sacrifice plant floorspace, time and energy to squeeze value from the acid. Management is often faced with the dilemma of allowing “the tail to wag the dog” — sacrificing speed of production over waste minimization. Recovery of the acid addresses that conundrum. Most of the commonly used and proven technologies are described below.

**Vacuum evaporation.** Hydrochloric acid is a binary solution of HCl gas in water used extensively in steel pickling and mining. When the pickling action slows, the inhibited solution of acid, water and metal salt enters a vacuum evaporator (Figure 5) and is heated above the boiling point of HCl (108.6°C) and water. To maintain sensible heat and ensure a more efficient heat cycle, the forced circulation of the mother liquor generates a mild backpressure. With the rest of the process operating under vacuum, the pressurized and heated solution is sprayed into a separator, where both the water and hydrochloric acid co-flash to vapor. The remaining metal chloride salt solution concentrates as more spent acid enters the evaporator and the HCl/H<sub>2</sub>O solution is vaporized. When the metal salt’s specific gravity reaches the target, the metal chloride concentrate is pumped to a storage tank for transport to an end user.

The HCl/H<sub>2</sub>O vapor travels through a rectifier filled with acid-proof packing. A spray of water

descending over the top of the packing mixes with HCl vapor as it descends. The droplets increase in acid concentration as they descend and approach the 20% HCl azeotrope. The remaining water vapor passes upward through the packing and into a water-cooled condenser. The vapor condenses into reusable process water. The rectification process results follow the McCabe-Thiele equation.

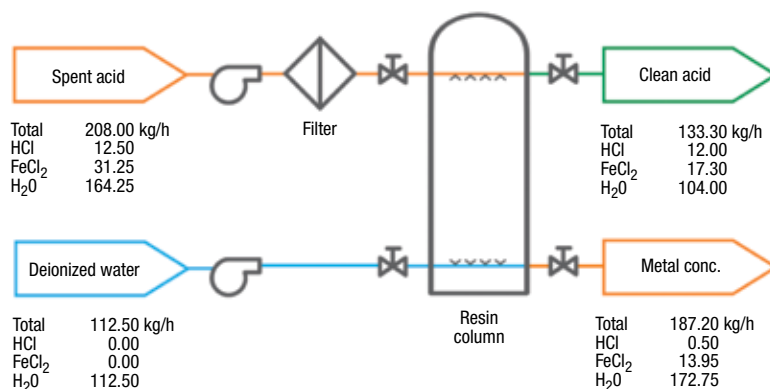
For efficiency, evaporative recovery operates under partial vacuum. This depresses the boiling point of HCl from 108.6°C (227°F) to less than 80°C (175°F). Vacuum processing lowers the energy consumption, cost of construction and potential for chemical excursions.

All three major streams from the vacuum evaporator are re-used or repurposed. The final acid concentration ranges between 15 and 20% — well within most pickling and leaching parameters. If acid concentrations are required above the 20% azeotrope, the addition of

a dual-pressure distillation section would be required. In steel pickling or leaching, the iron chloride concentrate captured has commercial value in water treatment, odor suppression and as a precursor to the production of ferric chloride. Processing one ton of spent acid requires approximately 22 therms of natural gas to produce the required steam.

**Acid sorption (retardation).** This technology is a sorption process by which acid bonds to a special resin inside a packed “short bed” column, while allowing the metal chloride and water to pass through. When the resins have reached maximum acid loading, the column is eluted using water to recover the absorbed acid on a batch basis. The process flow diagram (Figure 6) follows a near spent HCl solution from a steel pickling plant. The recovered acid stream contains residual iron and water. The waste metal concentrate is nearly devoid of acid but requires treatment as a liquid waste. The sorption process allows continuous use of the acid with low levels of metal contamination. Efforts over the past decade to minimize metal carryover into the recovered, clean acid have shown improvement. Selective capture and reuse of different fractions of the eluate tend to lower the waste and enrich the recovered acid product.

**Diffusion dialysis.** This membrane process (Figure 7) operates under the diffusion principle, where high concentrations of molecules spontaneously travel to areas of lower concentration. Eventually the con-



**FIGURE 6.** This acid sorption process processes 5 m.t./d of spent HCl



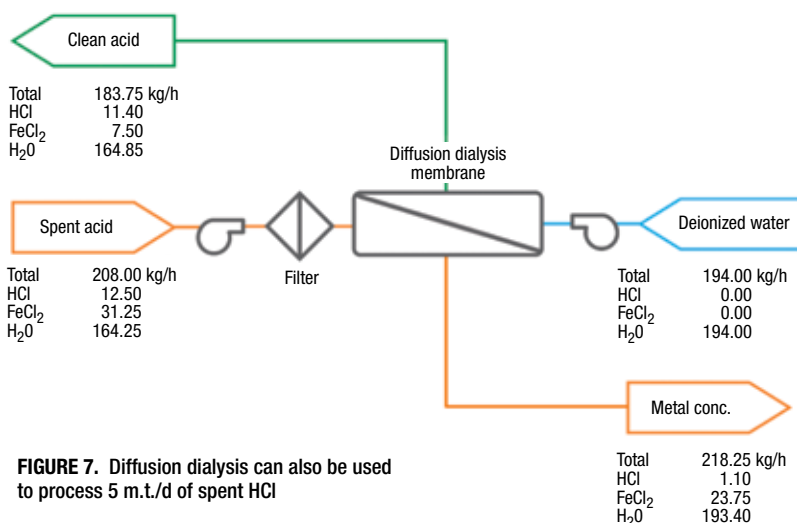


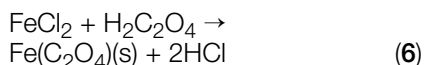
FIGURE 7. Diffusion dialysis can also be used to process 5 m.t./d of spent HCl

centration approaches equilibrium. Dialysis deals with the rate of molecular movement through a semi-permeable membrane. Diffusion dialysis uses an anion exchange (positively charged) membrane as the semi-permeable barrier. Although also positively charged, acid acts more like a small protonated water molecule and passes through the membrane at an order-of-magnitude-higher rate than the much larger metal salts. Clean water (dialyzate) is fed on the permeate side of the membrane to absorb acid passing through the semi-permeable membrane surface. By passing in a countercurrent manner, the acid concentration increases as it passes across a long, serpentine path toward saturation. Diffusion dialysis is a continuous process that depends on excellent mass transfer and multi-layered stacks of membranes. The membranes do not completely reject the metal ions but greatly retard their concentration. The membranes may be impacted by fouling and concentration polarization. Prefiltration and process control are very important to process efficiency and membrane longevity.

#### Acid replacement with oxalic acid.

Recent attempts to recover hydrochloric acid by reacting the spent acid with oxalic acid have shown some promise. Although the use of oxalic acid with iron-laden acid leach in the mining and minerals industry is not novel, use of the organic acid to treat and recover spent hydrochloric

acid is. The basic reaction seems to be the following:



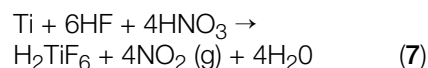
This batch process adds a measured amount of oxalic acid powder to a mixing reactor filled with an aliquot of the pickling bath. After a few hours of reaction, the relatively insoluble ferrous oxalate dihydrate forms as a coordination polymer that can be filtered from the slurry. The iron (II) oxalate forms a filter cake of small, mustard-colored solids that are usually landfilled. The filtered acid returns to the pickling process.

#### Mixed acid (HF/HNO<sub>3</sub>) recovery

The two largest industries using mixed hydrofluoric and nitric acid are stainless-steel manufacturing and titanium milling/etching. In the stainless-steel descaling process, oxides of iron, nickel and chromium (as well as other alloying elements) must be removed without causing underlying attack on the base metal. The combination of using oxidizing nitric acid as a passivation agent to protect the alloy surface, while at the same time applying hydrofluoric acid to remove scale from the surface, has worked well for over fifty years.

In titanium-milling operations, nitric acid dissolves some transient impurities from titanium, while the hydrofluoric acid dissolves the exposed titanium [Equation (7)]. The nitric acid mitigates hydrogen embrittlement while passivating the

titanium base metal. In both the titanium milling and stainless-steel cleaning and passivating processes, both the acid composition ratio and concentrations are important. The initial hydrofluoric acid concentration in a fresh solution rarely exceeds 10%.



The most commonly used technologies to recover HF/HNO<sub>3</sub> include: acid sorption, vacuum evaporation, pyrohydrolysis and nanofiltration. This highly corrosive acid combination presents a materials of construction challenge for recovery/recycle technologies. Since it etches glass as well as dissolves most metals, there are very few non-reactive containment materials. Thermoplastics such as polyethylene, polypropylene, PVDF and Teflon are effective storage and containment materials for both hydrofluoric acid and the mixed acid HF/HNO<sub>3</sub>. The added cost of these materials, however, is offset by the comparatively high cost of the acids.

**Acid sorption (retardation).** This resin method, previously discussed, has proven effective in recovering mixed acid for reuse. Even though the titanium, iron and other metal byproducts of the process cannot be economically recovered or reused, the high value of the acid justifies the capital and operating expense in many cases. The acidic waste is usually neutralized with lime before the sludge is filter-pressed for disposal.

**Vacuum evaporation.** Forced-circulation evaporation under vacuum has proven capable of recovering HF/HNO<sub>3</sub>, while producing a titanium fluoride salt with reclaim value. The materials of construction, operating temperature, and process control differ somewhat from the same technology used for recovering hydrochloric acid. The process uses the forced circulation co-flash principle to evaporate hydrogen fluoride, nitric acid, and water simultaneously. The rectifier section scrubs the acids, along with some water,

leaving a final stream of water condensate. The recovered metal concentrate can be crystallized or left in liquid form.

**Pyrohydrolysis.** Spray roasting technology also works on mixed acid. The capital cost is quite high, but the high cost of the acid can offset it in large stainless-steel production plants. Plants producing more than forty tons of spent acid each day might consider applying pyrohydrolysis. As with pyrolytic recycle of hydrochloric acid, the location of the recycling plant and air-quality restrictions should be studied.

**Nanofiltration and crystallization (experimental).** Acid recovery using nanofiltration membranes has been attempted many times over the past several decades. The three problems preventing the technology from advancing as a cost-effective solution have been the concentration limitation, flux rate, and the lack of corrosion-resistant high-pressure pumps and robust membranes. The ability to pass mixed acid through the membrane while sequestering the metal ions in a concentrate stream has been proven. However, as the metal ion concentration rises, the flux rate (throughput of recovered acid) diminishes. In tests at pressures up to 30 bars, the practical limit of metal ions in the spent acid was approximately 30 g/L.

The same acid-resistant nanofilter membranes pilot-tested for mixed acid recovery are used in copper recovery in sulfate leach solutions. The copper-sulfate-concentrate stream from nanofiltration is further processed in a crystallizer or electrowinning cell. Trial studies on mixed acid waste from stainless-steel cleaning lines in Sweden followed a similar approach — crystallizing the concentrate from the nanofiltration step and reprocessing the post-crystallization filtrate. Crystallization of the iron fluoride hydrate ( $\text{FeF}_3 \cdot 3\text{H}_2\text{O}$ ) proved difficult in the test trials, with the nickel and chrome hydrates even more so. The copper sulfate recovery from the concentrate of the membrane process was much simpler by comparison.

Microfiltration of the spent acid in

the first step of recovering the acid was found to be imperative. Ion-selective membranes and sorption resins require protection against incursion from solids and oil that cause blinding and fouling without excellent precleaning steps. In the case of filtration of the hydrofluoric/nitric mixed acid feed, some installations employ silicon carbide microfilters and corrosion-resistant components to extract the larger molecular organics like oil, as well as any microsolids that blind surfaces and impact the hydraulic process.

### Final remarks

Environmental protection agencies have set goals and standards that guide us toward what we now call ZLD (zero liquid discharge). Meeting the environmental standards with minimum impact on the cost of production has become the challenge to industry. Over the past fifty years, many recovery technologies have been applied to the mining, milling and metal-finishing industries to cost-effectively recover or recycle their most expensive and dangerous waste — acid. Advancements in materials and automation over that half-century have greatly reduced the capital cost of recovery and recycle, while also bringing operating costs into line. This challenge will continue.

Scientists and engineers work every day to create more cost-efficient, user-friendly processes. As we advance toward ZLD, innovations in recovery technologies create co-products rather than waste products. In many cases, the co-product revenue exceeds the operating cost. The goal has not changed, but the tools to reach it have advanced. As famed American-football coach Vince Lombardi once surmised, "Perfection is not attainable, but if we chase perfection, we can catch excellence." ■

*Edited by Gerald Ondrey*

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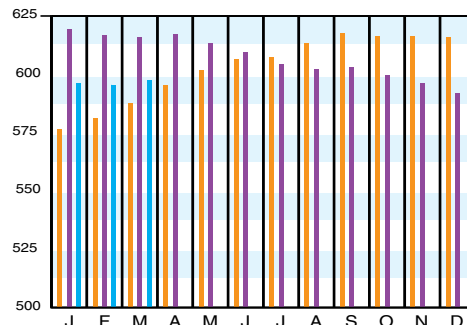
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## CHEMICAL ENGINEERING PLANT COST INDEX (CEPCI)

(1957-59 = 100)	Mar. '20 Prelim.	Feb. '20 Final	Mar. '19 Final
CEIndex	598.3	595.2	616.0
Equipment	726.2	722.0	752.8
Heat exchangers & tanks	621.4	615.5	668.4
Process machinery	724.7	722.1	728.2
Pipe, valves & fittings	954.7	952.0	977.7
Process instruments	416.7	416.9	421.2
Pumps & compressors	1085.2	1083.6	1066.0
Electrical equipment	562.3	563.7	557.5
Structural supports & misc.	778.5	767.2	827.7
Construction labor	335.7	335.4	334.5
Buildings	595.1	590.7	599.8
Engineering & supervision	313.4	313.1	316.9

Annual Index:  
2012 = 584.6  
2013 = 567.3  
2014 = 576.1  
2015 = 556.8  
2016 = 541.7  
2017 = 567.5  
2018 = 603.1  
2019 = 607.5

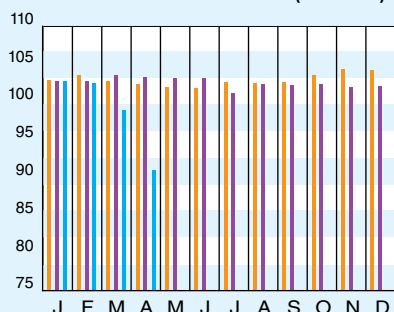


Starting in April 2007, several data series for labor and compressors were converted to accommodate series IDs discontinued by the U.S. Bureau of Labor Statistics (BLS). Starting in March 2018, the data series for chemical industry special machinery was replaced because the series was discontinued by BLS (see *Chem. Eng.*, April 2018, p. 76-77.)

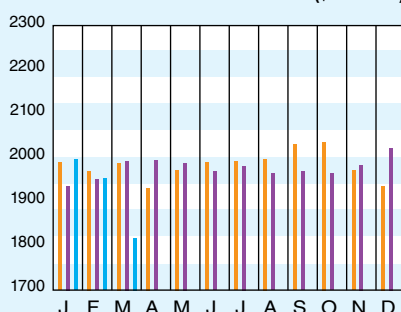
## CURRENT BUSINESS INDICATORS

	LATEST	PREVIOUS	YEAR AGO
CPI output index (2012 = 100)	Apr. '20 = 90.0	Mar. '20 = 99.2	Apr. '19 = 102.2
CPI value of output, \$ billions	Mar. '20 = 1,811.5	Feb. '20 = 1,987.5	Mar. '19 = 2,024.6
CPI operating rate, %	Apr. '20 = 67.0	Mar. '20 = 73.7	Apr. '19 = 76.5
Producer prices, industrial chemicals (1982 = 100)	Apr. '20 = 211.9	Mar. '20 = 238.3	Apr. '19 = 259.0
Industrial Production in Manufacturing (2012 = 100)*	Apr. '20 = 85.5	Mar. '20 = 99.1	Apr. '19 = 104.3
Hourly earnings index, chemical & allied products (1992 = 100)	Apr. '20 = 191.1	Mar. '20 = 187.1	Apr. '19 = 185.2
Productivity index, chemicals & allied products (1992 = 100)	Apr. '20 = 100.7	Mar. '20 = 99.2	Apr. '19 = 96.7

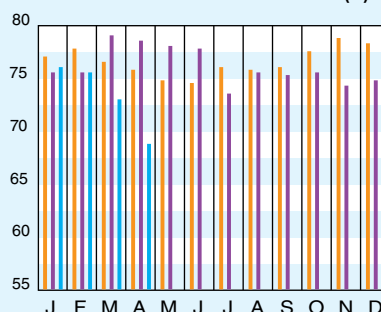
### CPI OUTPUT INDEX (2000 = 100)†



### CPI OUTPUT VALUE (\$ BILLIONS)



### CPI OPERATING RATE (%)



\*Due to discontinuance, the Index of Industrial Activity has been replaced by the Industrial Production in Manufacturing index from the U.S. Federal Reserve Board.

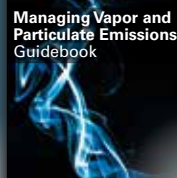
†For the current month's CPI output index values, the base year was changed from 2000 to 2012

Current business indicators provided by Global Insight, Inc., Lexington, Mass.

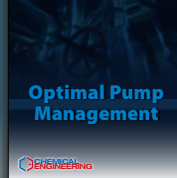
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## CURRENT TRENDS

The preliminary value for the CE Plant Cost Index (CEPCI; top) for March 2020 (the most recent available) increased compared to the previous month's value, with the values of the four major subindices (Equipment; Construction Labor; Buildings; and Engineering & Supervision) comprising the CEPCI all seeing small increases. The current CEPCI value is 2.9% lower than the corresponding value from a year ago at the same time. Meanwhile, the Current Business Indicators (CBI; middle) saw steep declines in the April data for the CPI output index as well as the CPI operating rate. In addition, the CPI value of output index for March 2020 dropped by a margin that was larger than the normal monthly fluctuations.